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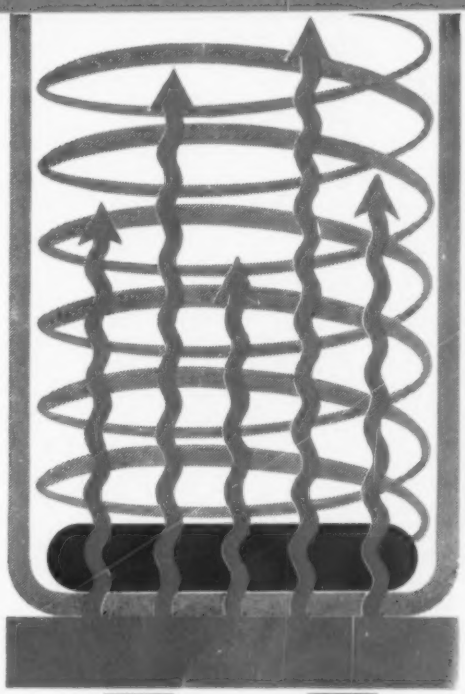

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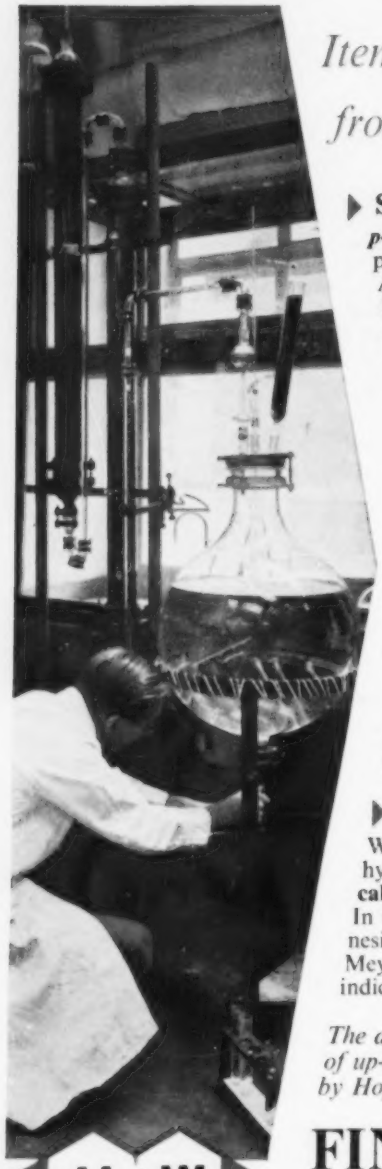

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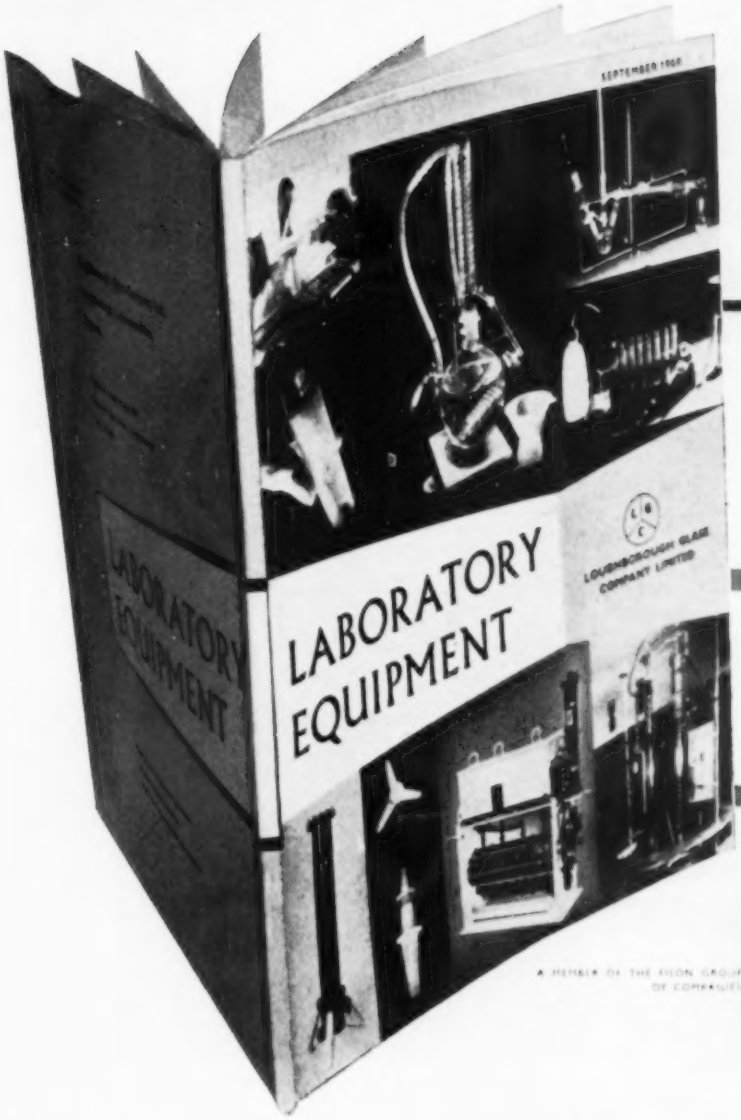
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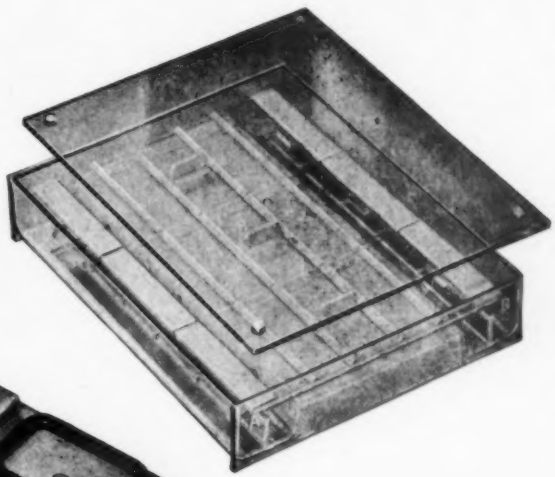

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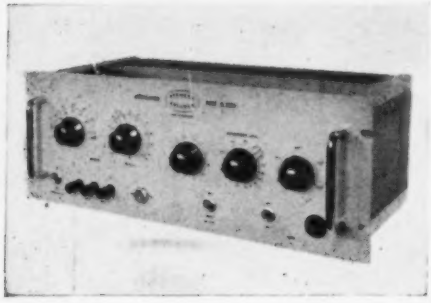

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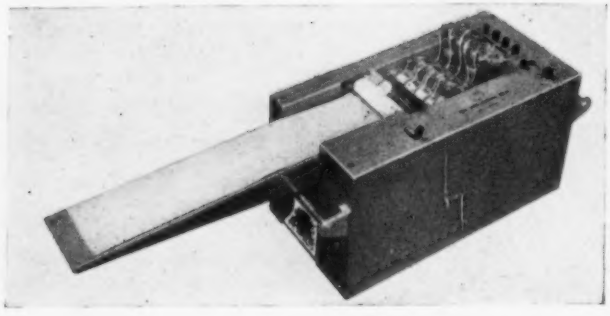

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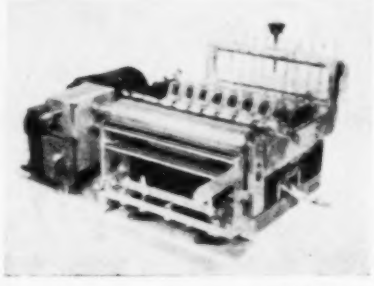

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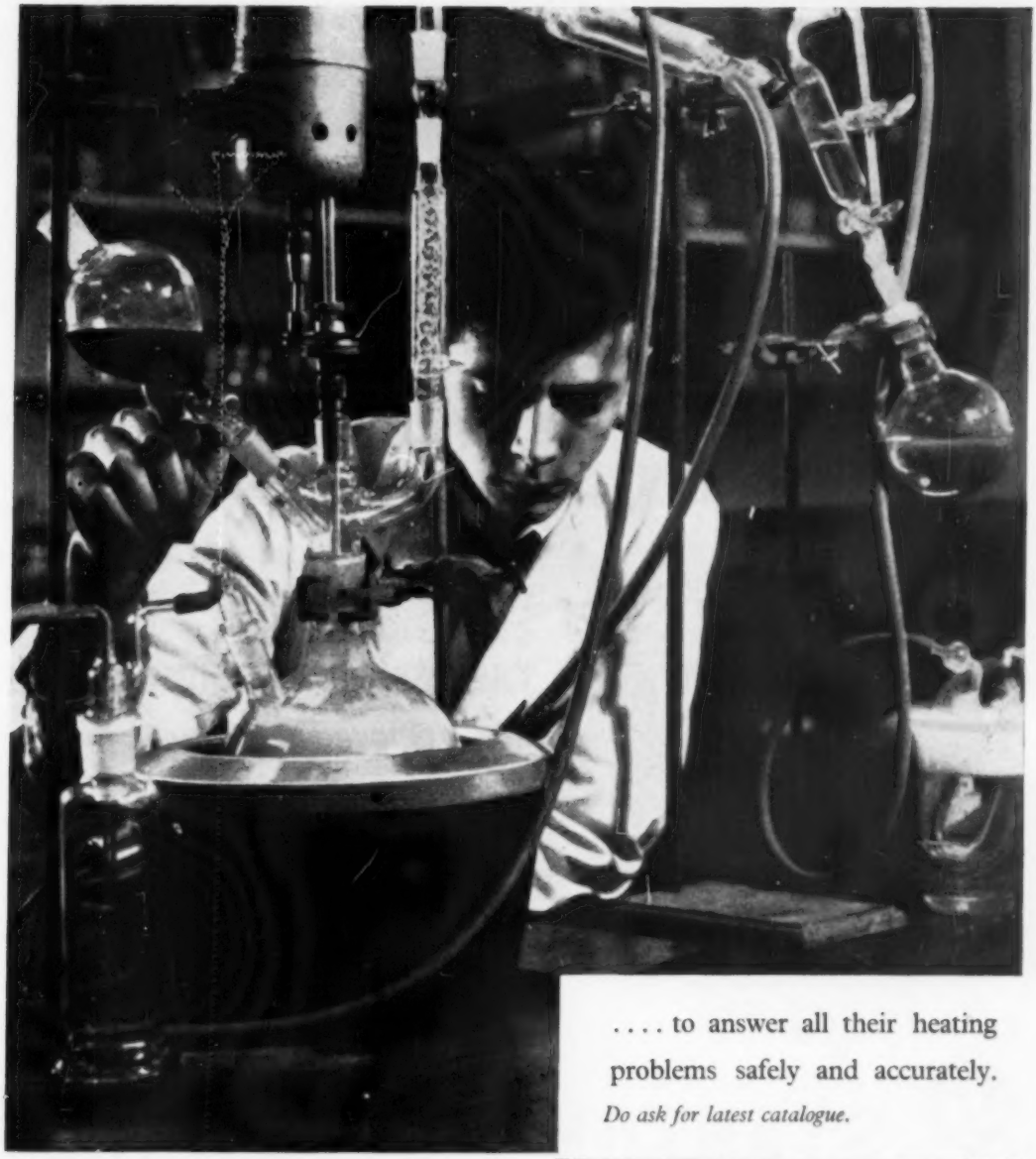

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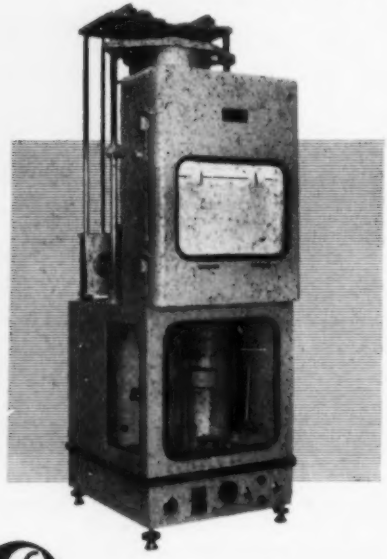

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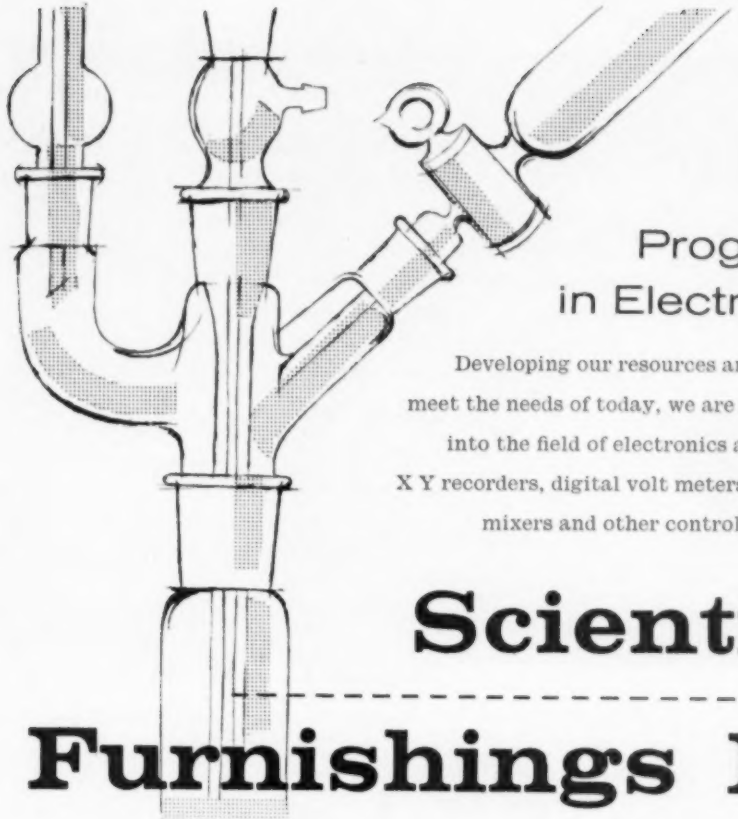

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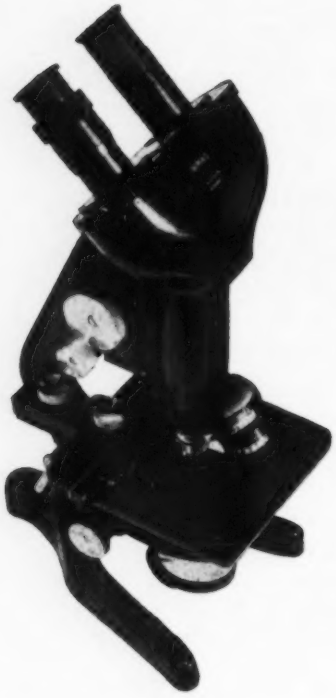

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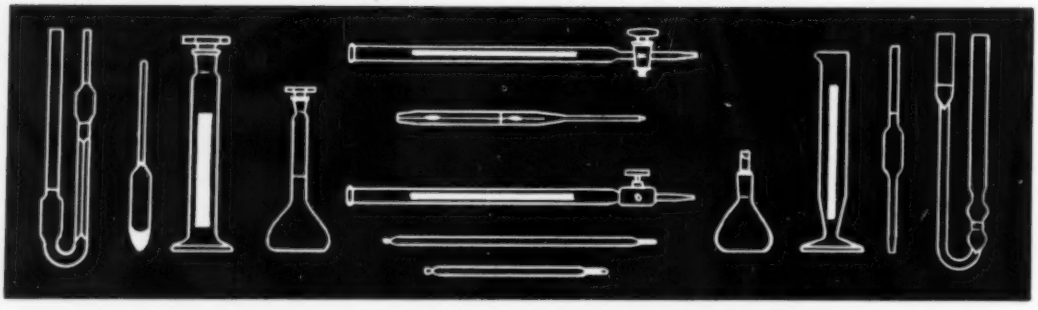

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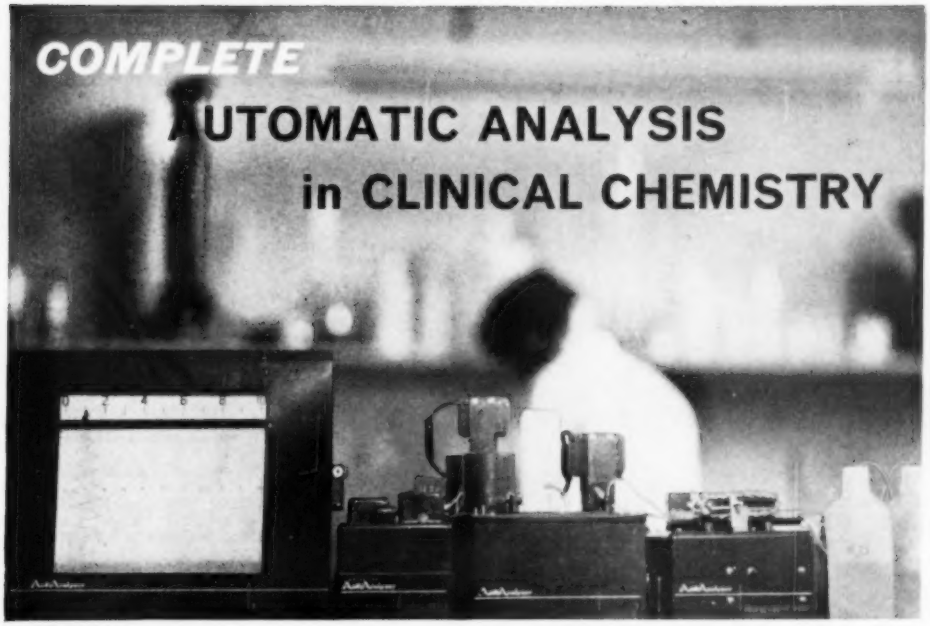

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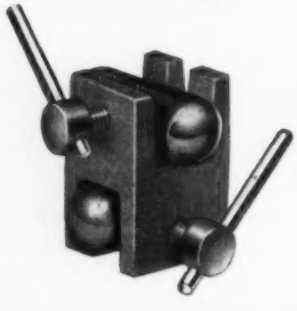

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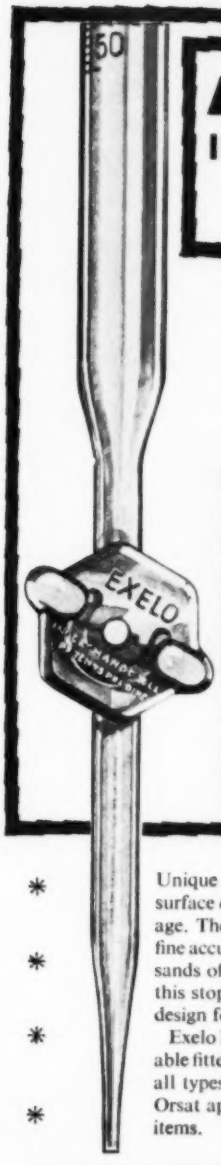

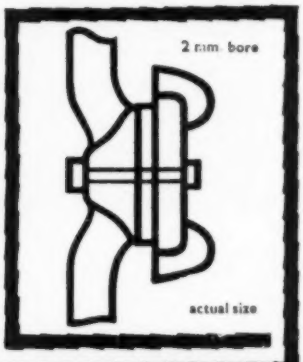

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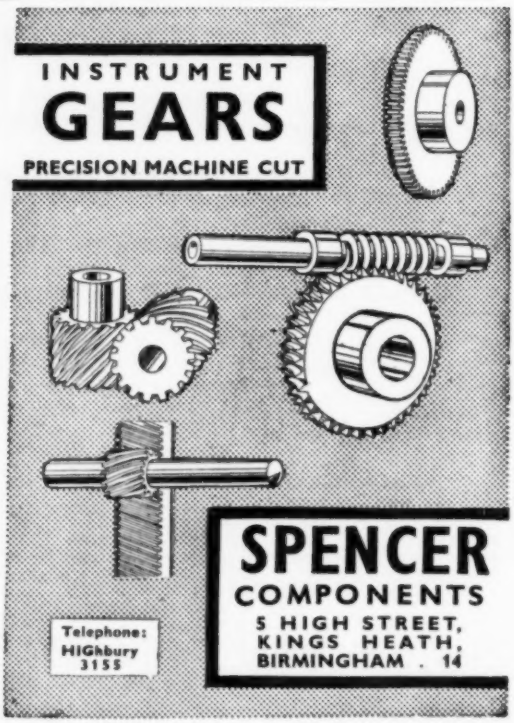

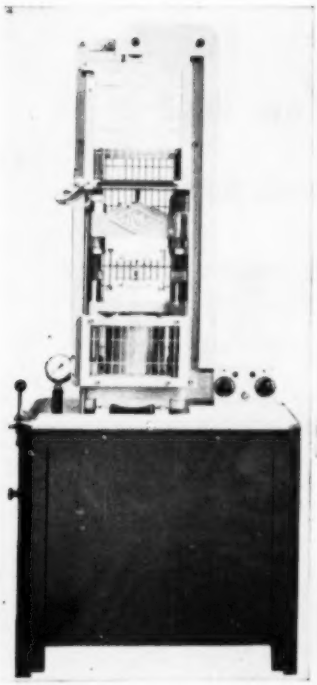

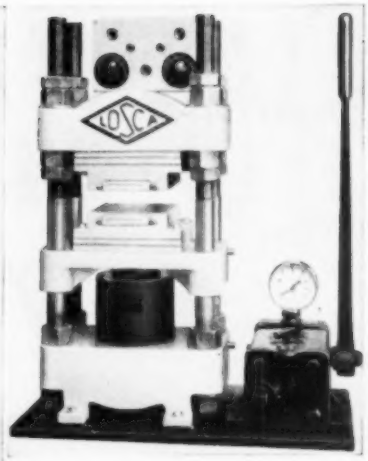

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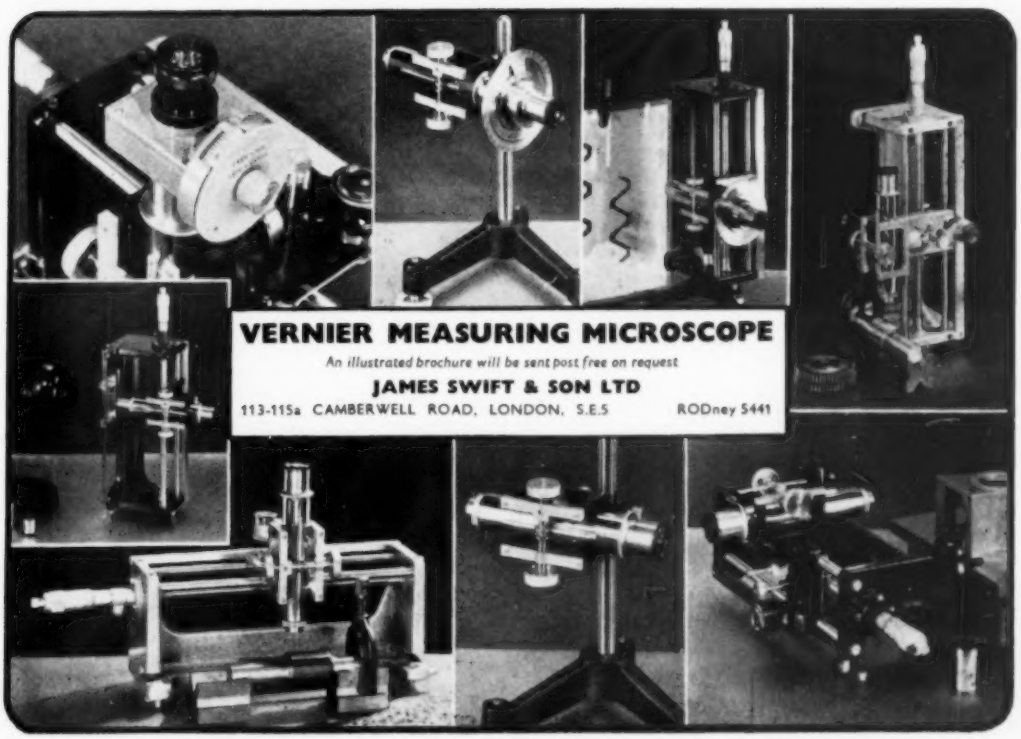

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EDITORIAL

First Things First

THE latest effort by those responsible for transport in this country has not unexpectedly aroused considerable criticism. The outstanding problems are to speed up traffic and reduce accidents. What contribution will be made by instituting compulsory examinations of cars over ten years old is difficult to say. So far as statistics on the causes of accidents can be relied on, it is admitted that these show that mechanical failure, even of older cars, are responsible for an insignificant proportion of accidents. We have a Road Research Laboratory in addition to Ministry of Transport officials, policemen and other officials concerned with the control of traffic, but every experienced motorist knows the causes of accidents. The most important are excessive speed at bends and cross roads, reckless overtaking and errors of judgment on the part of the driver. it is well established that some drivers are accident prone, but we have not seen any evidence that cars over ten years old are similarly affected.

It is surprising that a nation which is second to none in the fields of physics and engineering should be so backward and unimaginative in tackling its traffic problems. Admittedly the number of vehicles has increased enormously in the last few years but surely this means that there is all the more need for worthwhile measures. Tests on old cars, and traffic wardens for increasing the frequency with which motorists can be fined for parking, will not do much to solve our two main problems. The only steps we seem to take are merely devices to make more officials, more forms and more fines for the already harassed motorist.

A scientific approach to the problem would involve first the obtaining of reliable evidence on the causes of accidents and then consideration of the best means to eliminate the causes. As an example, we may discuss sudden changes of direction or stopping as a major cause of accidents in towns where high speeds do not come into question. The usual reason is that the driver, not knowing the town, sees only at the last moment a direction sign, or suddenly sees one which is inconsistent with the one last seen. It is not possible to blame any one person or authority for the chaotic, and sometimes stupid, system of road signs in this country, but it

would be far more intelligent to spend money on this sort of improvement than on the 'forms and officials' type of legislation. Any motorist returning from the continent will appreciate this point. There are some things that we would do well to adopt from our continental neighbours and the metric system and international road sign system are two good examples. Of particular importance are danger warning signs. We sometimes find strong warnings before a not dangerous bend or hill, and at other times a mere indication or none at all before a really dangerous one. One sometimes wonders if the people responsible for traffic control have ever driven a car in their lives.

If it is recognized that the major cause of accidents is the bad driver, surely the remedy is to deter bad driving, not by prison sentences which only add a further load to the over-burdened tax-payer, but by heavy fines and, for repeated offences, cancellation of the driving licence for long enough to punish the bad driver. The latter method would at least do a little to reduce traffic congestion.

Much could be done to ease traffic jams by instituting one-way systems and by publicizing less known routes. Even on bank holidays it is possible to travel with very little interference or hold-ups by careful selection of secondary roads only a short distance from a main highway which may be almost a solid mass of vehicles.

By far the cheapest and quickest way to accelerate traffic flow would be a considerable extension of one-way streets. Nearly 100 per cent of vehicles in large towns are there every day and the drivers know their routes very well. Until fly-overs are universal at all bad crossings we might at least try more one-way systems. Even reversible systems, i.e. for entering a town before 1 p.m. and leaving it after this time should at least be tried experimentally on a small scale. Even one carefully controlled experiment would be worth more than all the argument and theoretical discussions in the papers and elsewhere on any one suggestion.

The method by which it is decided which drivers to prosecute for parking is another mystery. The reason for choosing a car parked in a long line of cars in a straight road, where no obstruction is caused, and ignoring cars in the next road parked badly, or on corners so causing obstruction, is one that has so far eluded us. Another mystery is why one person can leave a broken-down car outside another person's house almost indefinitely, while a motorist may be prosecuted for parking his own car outside his own premises for only a short time. No one owns the highway, but it would be reasonable to permit some priority for the property owner and rate-payer.

Cars over ten years old on the road may be a nuisance because on average they are slower than newer cars, but they are not dangerous. Because of their slower speed and type of driver, they might be expected to be safer. If there is evidence that the older the car the more likely it is to cause accidents, why not start with tests for cars over 20 years old and gradually work downwards? This would at least reduce the ever expanding numbers of forms and officials.

International Geophysical Year Results

A course of 20 University Extension Lectures will be given at The School of Pharmacy, Brunswick Square, W.C.1, on Mondays at 6.30 p.m. beginning October 10, 1960.

The groups of three or four lectures on each of six topics in the programme of the International Geophysical Year will include sufficient of the fundamentals of the subject to enable non-specialists to appreciate the significance of recent work.

The following are details of the course:

1960

OCTOBER 10 — **Introduction**
C. A. RONAN, M.Sc., F.R.A.S.
The Secretariat, The Royal Society.

OCTOBER 17, OCTOBER 24, OCTOBER 31 — **Oceanography**
G. E. R. DEACON, C.B.E., D.Sc., F.R.S.
Director, National Institute of Oceanography.

NOVEMBER 7, NOVEMBER 14, NOVEMBER 21 — **Gravity**
W. BULLERWELL, B.Sc., Ph.D., F.R.A.S., F.G.S., A.Inst.P.
Geophysics Department Geological Survey and Museum.

NOVEMBER 28, DECEMBER 5, DECEMBER 12 — **Seismology**
T. B. H. HALL, B.Sc.
Exploration Division, British Petroleum Research Centre.

1961

JANUARY 9, JANUARY 16, JANUARY 23, JANUARY 30 — **The Ionosphere**
W. R. PIGGOTT, O.B.E., B.Sc.
Principal Scientific Officer, Radio Research Station.

FEBRUARY 6, FEBRUARY 13, FEBRUARY 20 — **Cosmic Rays**
J. J. QUENBY, B.Sc., Ph.D., A.R.C.S., D.I.C.
Physics Department, Imperial College.

FEBRUARY 27, MARCH 6, MARCH 13 — **Geomagnetism**
R. G. MASON, Ph.D., A.R.C.S., D.I.C.
Geophysics Department, Imperial College.

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The Deputy Director (Extension),
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THE ANALYSIS OF SODIUM, POTASSIUM AND CALCIUM, IN BIOLOGICAL MATERIALS, BY THE 'EEL' FLAME PHOTOMETER,

with Algebraic Corrections for Mutual Interference Effects

by I. F. Duthie, B.Sc., B.V.M.S., M.R.C.V.S. and I. McDonald, M.A.

Rowett Research Institute, Bucksburn, Aberdeen

Summary

Statistical analysis of data from solutions containing sodium, potassium, calcium, and phosphorus as orthophosphate at the various relative concentrations likely to be encountered in solutions prepared from biological materials, provide regression equations relating the extent of interference to the concentration of the interfering elements for the effects of potassium on sodium, calcium on sodium, calcium and phosphorus on sodium, sodium on potassium, and sodium and potassium on calcium. A proposed correction system is demonstrated.

Introduction

Flame photometry is a useful and rapid method of determining certain cations in solution, but the results for some cations may be biased due to the presence of other ions. Usherwood (1957) has discussed this question generally. For the 'EEL' flame photometer Puffeles and Nessim (1957, 1959) have described the general effects of sodium on potassium, potassium on sodium, calcium on sodium and potassium, ammonium dihydrogen phosphate on sodium and potassium, the separate effects on calcium of sodium, potassium, ammonium monohydrogen phosphate, and of sodium and phosphate together; and Hemingway (1956) has described the separate effects on calcium of sodium, potassium and orthophosphate. While this work demonstrated the importance of mutual interference effects in two element systems in certain conditions it was not directed towards providing corrections. No information on systems of more than two cations is available for the 'EEL' flame photometer.

Several techniques are available for limiting interference by physical or chemical means (West, Folse and Montgomery, 1950; Leyton, 1951; Hemingway, 1956; Schuhknecht and Shenkel, 1957; Bond and Hutton, 1959). These approaches can reduce but do not usually eliminate interference by particular ions, and residual effects together with the influence of unaffected elements must be still considered.

Empirical methods of correction which have been suggested by other authors (Bills, McDonald, Niedermeier and Schwartz, 1949; Severinghaus and Ferrebee, 1957; Burriel-Marti, Ramirez-Muñoz and Asuncion-Omarrementria, 1957) were not applicable to the wide range of observations arising in this laboratory in investigations entailing regular analyses of the sodium, potassium and calcium contents of the food, body fluids and excreta of sheep. A correction system intended to provide accurate estimates of these three elements from measurements on a single solution or on dilutions of it, was therefore required. This was derived from the results of a series of experiments with solutions which contained known quantities of sodium, potassium and calcium covering the required ranges of relative concentrations.

At the same time the effects of phosphate on measurements of sodium, potassium and calcium were investigated in further experiments with pure solutions to find out whether phosphate should be eliminated from test solutions.

Experimental

The information in Table I was used as a guide to the ranges to be used for interference solutions.

Our method was to take the cations one at a time, make them up in a number of concentrations which gave readings 40 per cent or more of full scale deflection and then to see how much these readings were changed when the other elements were also present singly or in combination with each other. It was expected that these

TABLE I

Typical concentrations of sodium, potassium, calcium, and phosphorus (as phosphate) in various materials

Material	*Sodium*	*Potassium*	*Calcium*	*Phosphorus*
	mg./100 g.			
Dried grass nuts	103	1880	560	206
Concentrate mixture	68	576	462	553
Sheep faeces	67	222	1680	1130
	mg./100 ml.			
Sheep urine	30	1140	12	652
,, whole blood	260	60	<10	<10
,, plasma	239	12	<10	<10

changes in meter deflections would be of two kinds, resulting either from a change in the emission intensity of the element being measured, 'radiation interference', or from emission by the interfering elements passing through the filter system, 'breakthrough'. In order to estimate the extent of the latter effects, measurements were made on solutions containing only the interfering element, or elements, and none of the element which was ostensibly being measured.

The equipment, the working conditions and the techniques used are described in some detail because factors such as the nature of the gas and the gas and air pressures (Woldring, 1953), the purity of reagents (Collins and Polkinhorne, 1952) and volumetric error undoubtedly affect interference and since difficulty has been experienced in interpreting or comparing the results of previous reports, where these details have been incompletely stated.

Instrument and Conditions.—An 'EEL' model A, flame photometer (Collins and Polkinhorne, 1952) equipped with the standard gelatin in glass filters for sodium and potassium, and interference filter for calcium was operated under carefully controlled ambient conditions. The drainage system was modified to improve the stability of instrument response by leading the drain tube vertically down through the floor of the casing and the bench. The free end of the tubing was attached to a liquid trap (White, 1952). The carbon track potentiometer of the instrument was replaced by 10,000 ohm, 4 W. and 500 ohm, 4 W. wire wound potentiometers in combination (British Electric Resistance Co. Ltd.). These acted as coarse and fine controls respectively.

The photometer is graduated from 0 to 100 in 50 divisions. Meter deflection is referred to in terms of scale units (s.u.) where 1 s.u. = 0·5 scale division. Individual observations were made to the nearest 0·5 s.u.

Gases and Pressure Regulation.—Compressed air and coal gas were used, both from cylinders (British Oxygen Gases Ltd.). A single, double stage regulator (British Oxygen Gases Ltd.) was fitted to each cylinder. Compressed air at 80 cm. mercury, was passed through a 5 l. Pyrex ballast bottle. The pressure was adjusted finally by the photometer needle valve to read 12 lb./sq. in. This pressure was selected since it permitted calcium estimation without complicating sodium and potassium estimations. The coal gas at 9 cm. mercury was regulated and stabilized by a gas bleed, a constant pressure water head and a ballast bottle (5 l.) inserted in the gas line so that gas was delivered to the instrument at an unvarying pressure of 17·5 cm. water. The pressure was adjusted finally by the instrument needle valve, during the aspiration of demineralized water, to obtain a cone size and appearance uniform between runs. Pyrex gas filters (porosity 1) were incorporated in both air and gas lines.

Glassware.—Jena, G 20, 34b volumetric flasks with plastic stoppers, and Jena, G 20 bulb pipettes were used exclusively in all experiments.

Salts and Acids.—The Johnson, Matthey and Co. Ltd. Specpure salts, sodium chloride, potassium chloride and calcium carbonate, were used exclusively. They were dried at 200°C. for 16 hours and stored under vacuum. Analar orthophosphoric, hydrochloric and nitric acids were used (Edgecombe and Hewitt, 1954).

Water.—Deionized water (Elgastat, Elga Products Ltd.) of resistance greater than 1,000,000 ohms was used in all the experimental work.

Calibration of Photometer

Four standard solutions containing 1·0 and 0·5 mg. sodium or potassium per 100 ml. were prepared by dilution (40 + 1960) of stock solutions containing 1·2711 and 0·6356 g. sodium chloride, or 0·9534 and 0·4767 g. potassium chloride per litre. Two standard solutions containing 5·0 and 2·5 mg. calcium per 100 ml. were prepared by dissolving 0·2497 and 0·1249 g. calcium carbonate in diluted hydrochloric acid (3 + 17), evaporating to dryness on the hot plate and making up to 2 litres. Four separate dilutions from each standard solution completed three series of ten calibration solutions, in 500 ml. volumes, containing respectively 0·1, 0·2 . . . . 1·0 mg./100 ml. of sodium or potassium, and 0·5, 1·0 . . . 5·0 mg./100 ml. of calcium. Readings from these solutions, in random order, provided calibration charts for the conversion to concentrations of scale readings for unknown solutions.

Solutions for Interference Experiments

One or more solutions were made up for each element as described for the stock and standard solutions, so that the desired final concentration for any element in each analysis solution could be obtained without intermediary dilution by using bulb pipettes of not less than 5 ml. (± 0·01) in combination with volumetric flasks of not less than 100 ml. (± 0·06).

Setting of Photometer

When the instrument was used, whether for calibration, interference tests, or with unknown solutions, the samples were assembled in lots of five or less. For interference trials each lot included the reference solution of the element being measured. Before each lot was read the zero was set with water and the sensitivity was adjusted to give a deflection of 100 s.u. with 1 mg./100 ml. sodium or potassium, or 5 mg./100 ml. of calcium. Readings were made with the 0·5 mg./100 ml. sodium or potassium, or the 2·5 mg./100ml. calcium solutions, respectively, to check that the calibration did not alter.

Results

The following regression equations have been obtained to relate the difference in scale readings of analysis elements obtained with and without the interfering element or elements to the concentrations of the latter in mg. per 100 ml. In presenting these equations we use the notation that, for example, $D_{K/Na}$ represents the effect of potassium, and $D_{Ca,P/Na}$ the joint effect of

calcium and phosphate, each on the scale reading for sodium. The ranges of concentrations covered by the experiments are given in mg./100 ml. with each equation.

I $D_{K/Na} = 0{\cdot}93 \log_{10}$ (mg. K/100 ml.) + 1·7
Na: 0·4, 0·6, 0·8
K: 0·025, 0·05, 0·1 . . . 51·2

II $D_{Ca/Na} = 1{\cdot}23$ (mg. Ca/100 ml.) + 0·1
Na: 0·0, 0·4, 0·6, 0·8
Ca: 0·25, 0·5, 1·0 . . . 16·0

III $D_{Ca \cdot P/Na} = 0{\cdot}062$ (Scale reading for Ca) + 0·15
Na: 0·0, 0·4, 0·6, 0·8
Ca: 0·25, 0·5, 1·0, 2·0, 4·0
P: 0·0, 0·5, 1·0, 2·0, 4·0

IV $D_{Na/K} = 0{\cdot}4 \log_{10}$ (mg. Na/100 ml.) + 0·9
K: 0·4, 0·8
Na: 0·1, 0·2, 0·4, 0·6, 0·8, 2·0, 4·0 6·0, 12·0

V $D_{Na \cdot K/Ca} = A + B$
where
A = 0·375 (mg. Na + mg. K/5)/100 ml. + 0·18
B = (mg. Ca/100 ml.) [0·3-0·015 (mg. Na + mg. K)/100 ml.]
Ca: 0·0, 1·0, 2·0, 4·0
Na: 0·0, 0·8, 3·2, 12·8, 51·2
K: 0·0, 1·6, 6·4, 25·6, 102·4

Equations I and IV represent 'radiation interference', but the other equations represent 'breakthrough' and still hold true in the absence of the measurement element.

The presence of phosphate had only slight effects on sodium and potassium measurements (a concentration of 8 mg. phosphorus per 100 ml. increased them on average by 0·3 and 0·7 s.u., and a concentration of 40 mg. phosphorus reduced them by 0·7 and 1·5 s.u. respectively), but depressed calcium readings to such a large extent that no reasonably precise correction was possible.

The residual standard deviations corresponding to the regression equations were all of the order of ± 0·5 s.u., based on from 12 to 70 degrees of freedom. This gives a measure of the precision of individual scale readings corrected for interference.

Table II shows the magnitude of the mutual interference effects, calculated from Equations I, II, IV and V, affecting measurements on phosphate-free solutions prepared from the samples in Table I.

Discussion

The Mutual Interference Effects of Sodium, Potassium and Calcium.—In general the effects are in agreement at least as regards direction with results described hitherto (Collins and Polkinhorne, 1952; Edgecombe

TABLE II

Magnitude of mutual interference effects after removal of phosphate in analysis of materials in Table I. Interference as % of correct measurement, in s.u.

Material	*Effect of potassium on sodium* (*Equation I*) *per cent*	*Effect of calcium on sodium* (*Equation II*) *per cent*	*Effect of sodium on potassium* (*Equation IV*) *per cent*	*Effect of sodium and potassium on calcium* (*Equation V*) *per cent*
Dried grass nuts	+4	+7	+½	+ 2
Concentrate mixture	+3	+8	+½	+ 2
Sheep faeces	+2½	+31	+1	+ 2
,, urine	+4	+½	—	+13
,, whole blood	+1	—	+1½	+45
,, plasma	+½	—	+2	+40

It is emphasized that the figures refer to the particular samples analysed and that the effects for some other samples are much greater, e.g. for urine and faeces from sheep in abnormal states.

and Hewitt, 1954; Hemingway, 1956; Puffeles and Nessim, 1957; Bond and Hutton, 1959), The figures in Table 2 are indicative of the magnitude of the effects and are not of general applicability, as quite large differences in composition occur from one sample to another, especially in urine and faeces. It has been stated that dilution minimizes interference effects. Over the narrow dilution ranges we employed for the analysis elements, this effect was absent for sodium, negligible for potassium, and of secondary importance for calcium, and so the concentration of the analysis element appears in only one of the regression equations (Equation V).

Effects on Sodium.—The results presented here agree with the hypothesis that the enhancement of the sodium emission by calcium is due mainly to the passage by the sodium filter of calcium emission (Porter and Wyld, 1955; Bond and Hutton, 1959). The effect of potassium, on the other hand, was greater than filter 'breakthrough' could account for, and followed a different law from that of calcium, being linearly dependent on the logarithm of the concentration of the interfering element instead of on the concentration itself.

Effects on Potassium.—The effects of sodium on potassium were of the same nature as the reverse one, but the relative proportions of sodium to potassium in many materials are such that the correction will often be negligibly small. There was no detectable effect of calcium.

Effects on Calcium.—The effects of sodium and potassium on calcium each depended linearly on their respective concentrations, were independent and could largely be accounted for as filter 'breakthrough'. As the calcium concentration increased, however, the effects became greater or less according as the sum of the sodium and potassium concentrations in the analysis solution was less than or greater than about 20 mg./100 ml. This secondary effect suggests the presence of 'radiation interference' in addition to 'breakthrough'.

In general the effects on calcium will be important only when the concentration of calcium is low relative to those of sodium and potassium as in blood and urine.

Levels below which Interference is Negligible.—For each of the mutual interference effects of sodium, potassium and calcium, Table III shows the concentration of the interfering element which produces a difference of 0·5 s.u. For lesser concentrations the interference effects could usually be ignored.

TABLE III

Approximate concentrations of interfering elements in analysis solutions below which each interference effect is less than 0·5 *scale units*

Element under analysis	*Interfering element mg./100 ml.*		
	Sodium	*Potassium*	*Calcium*
Sodium	—	0·05	0·3
Potassium	0·1	—	+
Calcium	0·5	3·0	—

+ No 'radiation interference' found up to levels of 2 mg. Ca/100 ml., and no 'breakthrough' found up to levels of 16 mg. Ca/100 ml.

Applicability of the Equations

For 'EEL' flame photometers used with gas, or gas and air pressures, etc., differing from these described here, the numerical constants in the regression equations are likely to be different. It is suggested that work should be done with pure solutions, to check the extent of the difference and if necessary to recalculate the constants, before the correction method is applied.

The Effects of Phosphate

The only important effect of phosphate was to depress the calcium emission. Except when the calcium concentration exceeded twice the phosphate concentration, the depressive effect was so great as to make it impossible to estimate the former at all accurately, even when the latter is known. As a by-product of this effect the calcium 'breakthrough' with the sodium filter was also depressed, and in the presence of phosphate it is necessary to correct sodium by an expression depending on actual calcium emission rather than on true calcium concentration (Equation III).

It was concluded that whenever high levels of phosphate were likely to be present it would be easier to eliminate it from the solution than to correct for its presence, and that this course would be advisable for any level of phosphate when measurements of calcium had to be made. Low levels of phosphate could be tolerated when only sodium and potassium were to be determined.

Practical Application of the Interference Correction

Preparation of Samples.—Methods were devised which could be applied to any biological material to yield solutions free of the complications of organic substances, and of phosphate and sulphate, and similar in anionic form, anionic concentration and pH to the standards and made up solutions from which the interference corrections were obtained.

Suitable volumes or weights of the organic materials to be tested, e.g. about 3·50 g. grass nuts, in porcelain beakers, are dried on a hot plate and reduced to ash in a muffle furnace over 16 hours, at a temperature starting at 250°C. and rising to a maximum of 460°C. The ash is damped with diluted nitric acid (10 + 90), treated with 1 ml. concentrated nitric acid, heated to dryness on the hot plate (about 30 minutes) and then incinerated in the furnace for 15 minutes at 450°C. The materials

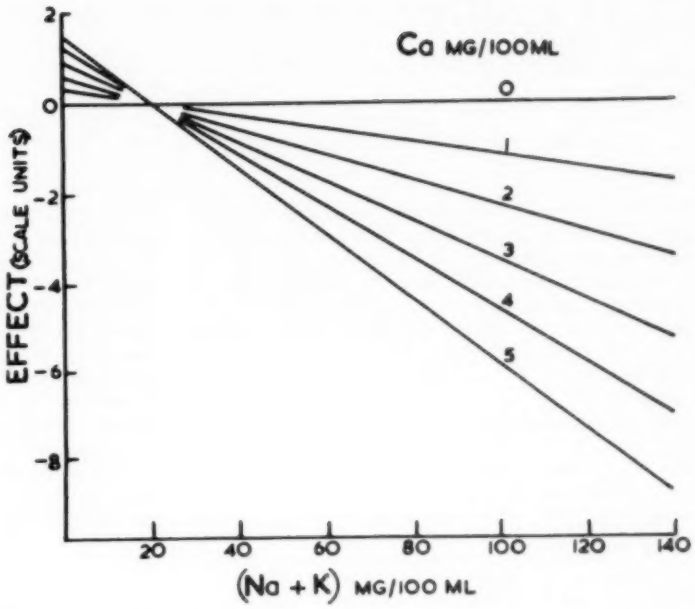

Fig. 1. The effect of sodium and potassium on the photometer reading for calcium: part B, equation V. The sum of the sodium and potassium concentrations is read off on the horizontal scale, and an ordinate is raised to a height determined by the uncorrected calcium concentration, interpolated among the lines on the chart. This ordinate is measured on the vertical scale to give the effect.

are then usually free of carbon, but, if not, the nitric acid and combustion treatments are repeated. The carbon-free ash is treated with 1 ml. concentrated hydrochloric acid and heated to dryness on the hot plate, then 5 ml. of diluted hydrochloric acid (1 + 99) are added and after digestion on the hot plate for 10 minutes the ash is extracted with hot water into 50 or 100 ml. volumetric flasks. If necessary, insoluble material is removed by filtration under reduced pressure through sintered glass crucibles (porosity 3) directly into the flasks. For each sample an aliquot of the extract is applied to a column of the anion-exchange resin Amberlite IR-45 (British Drug Houses Ltd.) in the chloride form and the eluate is collected in a 100 ml. volumetric flask. Photometric readings (40-100 s.u.) are made for at least one element on this solution, and for the other element or elements on dilutions of it.

Recovery Experiment to Test Accuracy and Reproducibility.—The overall effectiveness of the method, that is ashing and extraction, anion exchange, and correction system, was tested by recovery experiments. For example, a given weight of dried grass, a volume of solution containing sodium, potassium, calcium and phosphate equivalent to that in the dried grass sample, and half the weight of grass plus half the volume of solution, were taken through the entire procedure. Five replicates of each were done. The results from the mixed samples were consistent with the averages of those from the ashed grass and the pure solutions. The reproducibility of sodium, potassium and calcium estimations is indicated by the coefficients of variation which were respectively 1·1, 1·2 and 1·0 per cent. The absolute recoveries in terms of the known amounts of pure substance treated were 97·6 ± 0·5, 98·6 ± 0·5 and 102·4 ± 0·5 per cent for sodium, potassium and calcium respectively.

These seem satisfactory levels of accuracy and precision for many types of biological investigation.

Methods of Calculation.—One of the samples of dried grass nuts discussed in the foregoing section (the one used in Table 1) will serve to illustrate the arithmetical manipulations employed in correcting readings of sodium, potassium and calcium for mutual interference.

(*a*) The sodium and calcium readings from the column eluate, of which 100 ml. corresponded to 0·7 g. dried grass, were 86·5 and 80·0 s.u. respectively.

(*b*) The solution was diluted (5 + 95) and gave a potassium reading of 68·5 s.u.

(*c*) From calibration charts, and using the dilution factor, the (uncorrected) concentrations of potassium and calcium in the 'sodium' solution were therefore 13·22 and 4·0 mg./100 ml. respectively.

(*d*) From equations I and II, the corrections to the sodium reading for interference due to potassium and calcium were —2·8 and —5·0 s.u. respectively, giving a corrected reading of 78·7 s.u., equivalent to a concentration of 0·72 mg./100 ml.

(*e*) The concentration of sodium in the solution used for potassium estimation was therefore 0·036 mg./100 ml.

(*f*) From equation IV, the interference correction to the potassium reading, for sodium interference, was —0·4 s.u., giving a corrected potassium reading of 68·1 s.u., equivalent to a concentration of 0·657 mg./100 ml.

(*Concluded on page 732*)

A TECHNIQUE FOR BREEDING THE HOUSE FLY

Musca domestica L.

by A. Kirkwood

Ministry of Agriculture, Fisheries and Food, Central Veterinary Laboratory, Weybridge

SIMPLE techniques are essential when several species of arthropods are maintained in continuous culture, and for insecticidal tests it is also important to be able to produce on any one day large numbers of insects of similar age, size and sex ratio. It is the purpose of this paper to describe a simple method of breeding houseflies (*Musca domestica*) used at this laboratory for work with insecticides.

Adult Flies

The cultures are maintained in a constant temperature room at 25 ± 0·5°C. and 65-70 per cent relative humidity. Under these conditions the flies complete their development in 12 days. The adults are held in cubic cages 18 in. wide. These cages, made of mosquito netting and fitted with sleeves 12 in. long and 6 in. in diameter, are suspended by tapes at the four upper corners from taut wires stretched 2 ft. above the bench. They are easy to make, set up, wash and store. A small white enamel tray placed in the cage carries a dish of glucose powder and a pad of cotton wool soaked in fresh milk. Dried skimmed milk powder can replace the fresh milk providing water in cotton wool is offered (Basden, 1947), and a cage of 1,000 flies with sufficient water may be left unattended for five days.

At this laboratory a single cage of 1,000 flies is set up each week and discarded at the end of three weeks. The stock is correspondingly increased when flies are required for insecticide tests.

TABLE I

Weights of Pupae and Emergence of Flies from Cultures incorporating Milk and Yeast

No. of larvae per culture	*Culture No.*	*Milk and Yeast*				*Milk only*			
		Weight of pupae Mgm.		*% flies emerged*		*Weight of pupae Mgm.*		*% flies emerged*	
		Average wt. of 25 pupae	*Total average*	*%*	*Average %*	*Average wt. of 25 pupae*	*Total average*	*%*	*Average %*
500	1	26·4	26·1	72·0	80·6	24·4	24·0	57·6	67·3
	2	26·0		84·0		24·0		66·0	
	3	26·0		85·8		23·6		78·4	
750	1	25·0	27·4	70·6	80·4	23·0	24·2	50·6	66·6
	2	28·8		78·6		24·4		65·3	
	3	28·4		92·0		25·2		84·0	
1,000	1	27·0	27·0	74·0	71·5	24·0	24·7	64·3	69·8
	2	28·6		65·3		25·0		68·1	
	3	25·6		75·2		25·2		77·0	
1,500	1	23·7	24·3	73·4	77·2	22·2	20·9	68·6	64·7
	2	24·0		79·6		17·8		65·4	
	3	25·2		78·5		22·2		60·2	
2,000	1	24·0	23·8	66·2	63·2	20·4	17·9	42·6	52·7
	2	24·4		51·2		17·0		52·4	
	3	23·1		72·1		16·4		63·0	
2,500	1	24·0	23·3	60·0	64·5	18·0	17·6	54·8	61·5
	2	24·0		67·2		18·8		63·6	
	3	24·0		66·4		16·0		66·0	

Larval Cultures

Flies will readily oviposit on to a milk pad and eggs for stock culture should be taken from the two youngest cages, that is, from flies 1-14 days old, as the fecundity of a fly rapidly decreases after the tenth day when 65 per cent of the eggs will have been laid (Wilkes, 1948). For critical tests we prefer to take eggs from flies 1-7 days old in accordance with the standard set by the Chemical Specialities Manufacturers' Association (C.S.M.A.) of America which require for its standardized test that eggs shall be taken from flies not more than eight days old (Anon., 1960).

Flies used for critical tests must be of a standard age, size and sex ratio. Overcrowding of cultures will delay pupation (Smith, 1951) and stunted flies are produced. The abnormality is not passed on to the next generation (Basden, 1947) so that these flies, while quite unsuitable for standardized insecticide tests, can still be used for breeding purposes. The susceptibility to some insecticides varies with the sex of the species and as male flies tend to emerge before the females (Millar and Simanton, 1938), to delay pupation and hence emergence of one of a batch of cultures may upset the sex ratio. Overcrowding of cultures must obviously be avoided, and this can be done using a volumetric method in which the required number of eggs are taken in a given volume of water. Moreland and McLeod (1936) have described such a method using portions of 5 ml. graduated pipettes in which eggs were sedimented in water. In this laboratory eggs laid on the milk pad are allowed to hatch, and we find that after some experience and using the culture medium described, an estimated number of larvae can simply be transferred from the milk pad to a culture on the point of a scalpel.

The larvae are cultured in sweet jars 10 in. high and 4 in. wide. Various culture media have been described incorporating bran, grass meal, malt and yeast. Hafez (1948) listed some of these and has himself described a simple technique using cotton wool and milk only. In this laboratory we have for the past 12 years used an extract of yeast* with milk, and find, as shown in Table 1, that better results are obtained than if milk only is used. Approximately 20 g. of a commercial extract of yeast is sandwiched in 30 g. of absorbent cotton wool which is then placed in the jar and soaked with about 300 ml. of milk. Fisher and Jursic (1958) used dried milk and water in a similar medium incorporating dried yeast. An estimated 1,000 eggs or first instar larvae are then placed on the medium and the jar closed with a double layer of muslin held by two elastic bands. An inch of sawdust is added on the fourth day to provide a substrate for pupation.

The pupae can be separated by sieving or by tipping them on to an inclined surface down which they will roll, while the sawdust is being removed by a blast of air from an electric fan. Goodhue and Linnard (1950) used an air separator apparatus and Incho (1954) improved the technique by adding vermiculite to the medium. For routine purposes, however, the jars are placed in a cage and can be left there until the cage is discarded.

*Yeastrel from Brewers' Food Supply Co. Ltd.

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The Beilby Medal and Prize, 1961

Since 1930, at intervals of one or more years, awards have been made by the Administrators of the Sir George Beilby Memorial Fund, representing the Royal Institute of Chemistry, the Society of Chemical Industry and the Institute of Metals. Sir George Beilby had been President of each of these three bodies, and they jointly sponsored the appeal for subscriptions whereby the Fund was raised as a memorial to him after his death in 1925.

The awards are made to British investigators in science in recognition of independent original work of exceptional merit, carried out continuously over a period of years and involving the development and application of scientific principles in any field related to the special interests of Sir George Beilby, i.e. in chemical engineering, fuel technology or metallurgy, in their modern interpretations. The awards are intended as an encouragement to younger men and women (preferably under age 40) who have done distinguished work of practical significance in any of these fields.

In 1958 two awards, each of 150 guineas, were made. It was then agreed that no further award would be made before 1960.

The Administrators have decided that henceforth each award shall consist of a gold medal as well as a substantial sum of money, and shall be known as 'The Beilby Medal and Prize' and specified as being 'For Advancement in Science and Practice'. Such an award will be offered at intervals of two years, but more than one may be made on the same occasion if there are several candidates of sufficiently outstanding merit.

Consideration will be given in due course to the making of an award (or awards) from the Fund in 1961. Outstanding work of the nature indicated may be brought to the notice of the Administrators, either by persons who desire to recommend the candidate or by the candidate himself, not later than December 31, 1960, by letter addressed to The Convenor of the Administrators, Sir George Beilby Memorial Fund, The Royal Institute of Chemistry, 30 Russell Square, London, W.C.1.

The letter should be accompanied by nine copies of a short statement on the candidate's career (date of birth, education and experience, degrees and other qualifications, special awards, etc., with dates) and of a list of titles, with references, of papers or other works published by the candidate, independently or jointly. Photographic copies of these documents are acceptable. Candidates are also advised to forward one reprint of each published paper of which copies are available.

THE LABORATORY EVALUATION OF GANGLION BLOCKING AGENTS

by J. J. Lewis and T. C. Muir

Institute of Physiology, University of Glasgow

PART II—MEASUREMENT OF SYMPATHETIC AND PARASYMPATHETIC GANGLION BLOCKING ACTIVITY

Perfusion of the Superior Cervical Ganglion

The use of this preparation affords probably the most specific and almost certainly the most widely used means of measuring sympathetic ganglion blocking activity. The method described is that of Kibjakow, 1933[47], who used it to investigate physiological phenomena. It consists of perfusing the superior cervical sympathetic ganglion of the cat with oxygenated Locke's solution and recording the contractions of the nictitating membrane induced by electrical stimulation of the preganglionic fibres of the cervical sympathetic chain.

The animal was anaesthetized by means of chloralose, the skin covering the neck cut away from the top of the sternum to the apex of the mandible and the fascia covering the trachea divided at the midline. An aneurysm needle carrying a strong linen thread was passed between the muscles of the neck and then round the trachea. A transverse incision was made in the latter and by means of forceps holding the cut edge of the partially severed trachea, a cannula inserted and tied in place. The head of the animal was rigidly fixed by means of string attached to a brass rod, passed between the jaws. The ends of the rod were then clamped firmly in place and supported on uprights fixed to the sides of the operating table. By means of a fine linen thread passed through the mid-point of its margin, the nictitating membrane of one eye was pulled forward and outward to make approximately a 30° angle with the long axis of the cat. The thread was led by means of pulleys to a frontal point writing lever and contractions of the nictitating membrane recorded on a smoked drum surface.

The fascia covering the transverse vein which joins the two external jugular veins was then divided and the vein doubly ligated and cut. By means of blunt dissection and antero-lateral muscles of that part of the neck laying above the common carotid artery were divided on one side and the artery exposed. The course of the common carotid artery was followed upwards to the point of its division into the external and internal branches. It was carefully freed from connective tissue and adjoining nerve trunks and small blood vessels leaving it carefully ligated and cut. A ligature was then placed around the cardiac aspect of the common carotid artery and a second ligature placed distal to the point from which arises a group of small arteries which supply the superior cervical ganglion. The artery was cut at this point (Fig. 7). All branches of the artery, except those leading to the ganglion itself, were tied off. Similarly several small branches of the internal jugular vein were tied off leaving intact only the internal jugular vein together with a small vein which joins it from the ganglion; this vein carried the perfusate from the ganglion. The internal jugular vein itself was doubly ligated as shown in Fig. 7. The common carotid artery and the internal jugular vein were then cut and cannulated. By this means a complete two-way perfusion (outflow and inflow) of the ganglion was achieved while the composition of the perfusate could also be determined. The cervical sympathetic chain was now dissected from the vagus and a fine cotton thread tied tightly round it as low down in the neck as possible; it was then cut at a point above the ligature. The artery

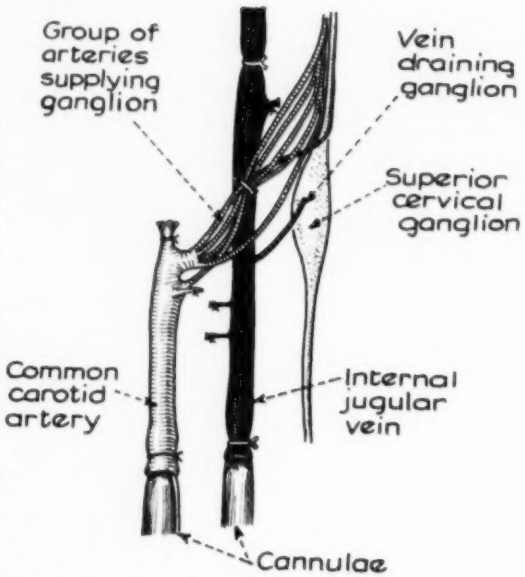

Fig. 7. Perfusion of the superior cervical ganglion of the cat. After Kibjakow[47] by kind permission of the publishers (Springer-Verlag, Heidelberg).

Part I of this series was published in the June, 1960 issue of LABORATORY PRACTICE.

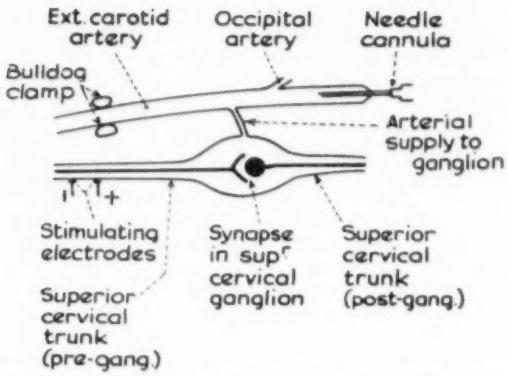

Fig. 8. Administration of drugs by close intra-arterial injection into the cat superior cervical ganglion. After Paton and Perry[50] by kind permission of Prof. W. D. M. Paton and Prof. W. L. M. Perry and the editors of the Journal of Physiology.

cannula was connected to a Mariotte bottle containing oxygenated Locke's solution at 37-38° and perfusion began.

The cervical sympathetic chain was stimulated electrically and contractions of the nictitating membrane recorded. Stimulation was continued for two to three minutes and followed by a period of rest of one to two minutes. Supra-maximal stimulation of the order of 10 impulses per sec. were employed. Feldberg and Gaddum[48] modified the composition of the perfusion fluid by reducing the amount of potassium chloride. It was found by Kibjakow[47] and Feldberg and Gaddum[48] that cannulation of the internal jugular vein in the cat was difficult due to its very small diameter, and the latter authors used instead the small vein which emerged from the vertebral column through the atlantal foramen and which joined the jugular vein near the superior cervical ganglion. These workers also stressed the importance of filtering the perfusion fluid. In their experiments the artery cannula was warmed by means of an electrically heated wire wound around it and the temperature and rate of flow of the perfusing fluid controlled at 40° and 0·1-0·3 ml. per min. respectively.

Both Kibjakow[47] and Feldberg and Gaddum[48] used this technique to investigate fundamental physiological phenomena for which a knowledge of the composition of the perfusate was required. This information is not essential for the estimation of ganglion blocking activity. Consequently a modification of the original technique has been used by many workers investigating sympathetic ganglionic transmission and blockade. This involved intra-arterial injection of the drug without actual ganglion perfusion. A method employing this modified technique will now be described.

Paton and Perry[49,50] used essentially the method of Kibjakow[47] and left the venous blood supply intact. A needle cannula was inserted into the distal cut end of the external carotid artery and with the exception of the occipital artery (to allow a free flow of blood past the ganglion) and those small branches to the ganglion itself all other branches of the common carotid artery were tied off. Recording electrodes were used to investigate potential changes in the ganglion itself and impulses were led to an amplifier and observed on an oscillograph. Retrograde intra-arterial injections could be made into the external carotid artery, a bulldog clip being placed proximal to the cannula while the injection was being made (Fig. 8). Perry (1953)[51] maintained the perfusing fluid constantly at body temperature by passing the inflow tube through the oesophagus from below upwards, so that its tip emerged at the point of insertion of the carotid artery cannula.

Instead of perfusing the superior cervical ganglion, intravenous administration of the drugs under investigation into the femoral or external jugular veins has been used to estimate sympathetic ganglion blocking activity.

After severing the preganglionic cervical sympathetic chain, contractions of the nictitating membrane were elicited by electrical stimulation of the cephalad end using square wave impulses at a frequency of 800-1,200 per minute at 8 to 15 volts, pulse width being 0·5-1·0 msecs. Stimulation reaches the nictitating membrane across the synapses of relays of ganglion cells in the superior cervical ganglion and any ganglion blocking activity of the drug is indicated by a reduction or elimination in the height of the contraction of the nictitating membrane.

Inferior Eyelid of the Anaesthetized Rat

Isola and Bacq, 1946,[52] investigated the innervation of the inferior eyelid of the cat and dog and demonstrated the sympathetic nervous control of these tissues. They also demonstrated the activity of certain drugs and suggested that this preparation could be used to study the properties of adrenergic nerves.

In 1956 Gertner[53] showed that the rat provided a sensitive means of studying the effects of drugs acting on smooth muscle and sympathetic ganglia utilizing the inferior eyelid preparation. His method is described below.

White rats weighing approximately 200 g. were anaesthetized by intraperitoneal injection of sodium pentobarbitone and the head immobilized by means of a horse shoe clamp.

The trachea was cannulated, and using a dissecting microscope the cervical sympathetic trunk dissected out tied peripherially and cut. The nerve was stimulated using silver-silver chloride electrodes, an isolation unit was necessary to prevent unwanted stimulation of the sympathetic trunk. A silk thread was tied through the lower eyelid and fixed to a mirror which was suspended by a fine taut wire. A narrow beam of light was focused on the mirror and the edge of the reflected light beam allowed to fall on a photocell. As the eyelid retracted, a proportionately greater amount of light reached the photocell and this was amplified and recorded. Stimulation with a pulse width of 250 μ sec. for a period of five

seconds using supramaximal voltage was employed. Drugs were administered intravenously into the femoral vein. This method has been shown to be useful for assaying ganglion blocking drugs when only a small quantity of material was available.

Methods of Estimating Parasympathetic Ganglion Block

Inhibition of the Peristaltic Reflex.—Three types of muscle movement are shown by mammalian small intestine. These are: (1) pendular motion, the frequency of which is dependent on the size of the animal; (2) changes in tone, which are superimposed on the first type of movement, and (3) peristalsis. The peristaltic reflex is elicited by an increase of intraluminal pressure in the intestine and has the specific function of propelling the intestinal contents along the gut. It consists of two phases: a preparatory phase, associated with contractions of the longitudinal muscle of the intestine, followed by an emptying phase in which contraction of the circular muscle of the intestine takes place[54,56]. The contractions of the circular layer of muscle are controlled by the autonomic nervous system, while those of the longitudinal muscle layer are an inherent property of the muscle fibres themselves and remain unaffected by drugs which interfere with nervous structures in the intestinal wall.

Bülbring, Lin and Schofield[55] studied the anatomical and physiological features of the peristaltic reflex and described two separate types of neurones in the gut, unipolar or bipolar neurones and multipolar neurones. They also confirmed that Meissner's plexus was sensory in function, while Auerbach's was motor. The appearance of the peristaltic reflex in response to an increase in intraluminal pressure is due to stimulation of pressure-sensitive receptors in the intestinal mucosa. The nervous mechanism is controlled from within the wall of the intestine itself, and consequently the cutting of extrinsic nerve fibres does not influence the reflex. It is however affected by any interference with the sensory nerve fibres in the mucosa.

As the ganglia involved in the peristaltic reflex are probably parasympathetic in origin[56], inhibition of the reflex is a means of estimating the potency of drugs inducing parasympathetic ganglion blockade. The effects of local anaesthetics and curare-like drugs have also been estimated in this way and the literature has been recently reviewed by several workers[33,57-59]. The most commonly employed method of studying peristalsis is that of Trendelenburg[54] who used isolated pieces of guinea pig intestine. Guinea pig intestine was chosen because it shows smaller pendular movements and less variability in tone than other mammalian species. The animals were anaesthetized by subcutaneous injection of ethyl urethane and the intestine removed and kept in aerated Tyrode's solution at body temperature. A piece 7-10 cm. long was tied at the oral end and the thread attached to a writing lever. The caudal end was connected to a glass tube which was connected to a bottle filled with Tyrode's solution. The bottle could be raised or lowered as required to subject the intestine to a measureable, intraluminal hydrostatic pressure. The level of the liquid in the bottle was similar to that in the organ bath which contained the intestine. All air bubbles were removed from the system and the neck of the bottle connected by rubber and glass tubing to a float volume recorder connected to a ring lever writing on a smoked surface. By raising the bottle, intraluminal pressure was increased to a threshold value and peristalsis initiated. The pendular movements of the intestine were recorded at the same time by means of the lever attached to the oral end of the preparation.

Several modifications have been made to this method. Feldberg and Solandt[60] used rabbit intestine and killed the animals by a sharp blow on the back of the head followed by exsanguination instead of using anaesthesia. In the method described by Trendelenberg[54] the peristaltic reflex is readily fatigued and for this reason Feldberg and Lin[56] stimulated the intestine for short periods of time only. They also stressed the difficulty of

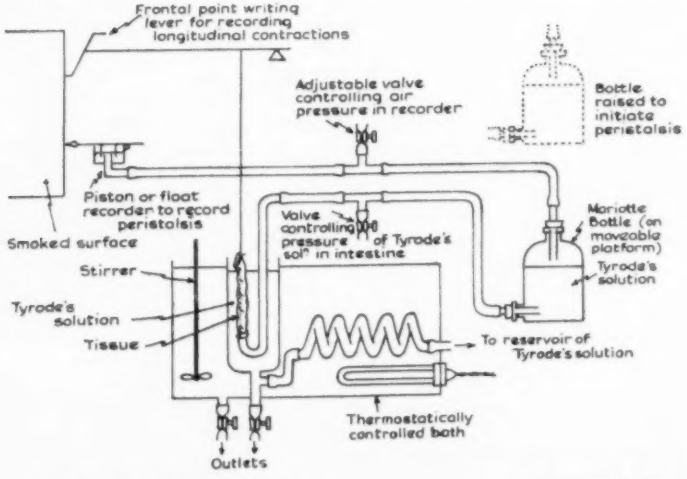
Frontal point writing lever for recording longitudinal contractions
Adjustable valve controlling air pressure in recorder
Bottle raised to initiate peristalsis
Piston or float recorder to record peristalsis
Smoked surface
Valve controlling pressure of Tyrode's sol^n in intestine
Mariotte Bottle (on movable platform)
Tyrode's solution
Stirrer
Tyrode's solution
Tissue
To reservoir of Tyrode's solution
Thermostatically controlled bath
Outlets

Fig. 9. Apparatus used to record peristalsis and longitudinal contractions in isolated intestinal tissues.

interpreting the results as the records obtained for true peristalsis and pendular type muscle contractions may be confused. Feldberg and Lin allowed the gut to remain suspended in the bath with the upper end open for half to one hour prior to commencement of the experiment, and during this period it was frequently rinsed with Tyrode's solution.

Edland and Gorbatschev[67,68], used essentially Trendelenberg's method and connected a T-piece to the caudal end of the intestine. One branch of the T was connected to a suction device by means of which the lumen could be rinsed during the experiment. These workers used rabbits anaesthetized by intravenous injection of urethane.

In the original method described by Trendelenberg[54] the fluid bathing the inside of the lumen could not be changed and consequently drugs could not be administered to the inner surface of the intestine without interfering with the recording. Methods to overcome this have been described by several workers who measured the movement of fluid along a loop of intestine *in situ*[65,66]. A method of recording the peristaltic reflex in response to automatic constantly recurring stimuli and which enabled drugs to be applied to both inner and outer intestinal walls was described by Bülbring and Lin in 1958[65]. Both ends of the piece of intestine were held rigidly and consequently no record of longitudinal contractions was obtained. This is overcome in the method described by Bülbring, Crema and Saxby[66] who suspended the piece of intestine longitudinally in the bath and connected the oral end to a J-tube and the caudal end to an inverted U-tube. The former was attached to a horizontal lever and recorded the logitudinal contractions of the intestine while the latter was connected to a float recorder and recorded peristalsis.

Numerous modifications to Trendelenberg's original apparatus have been made. Several volume recorders have been designed and developed varying in complexity from the simple bell jar type originally used by Trendelenberg[54] to a modern electro-magnetic device of the type described by Streeten and Vaughan-Williams[64]. Similarly various levers are in use for recording the longitudinal contractions. Details of these are to be found in the literature.

Fig. 9 shows an apparatus used in these laboratories for recording peristalsis in the rabbit and guinea pig ileum. This is a modification of Trendelenberg's original design. A typical tracing is shown in Fig. 10.

(*To be continued*)

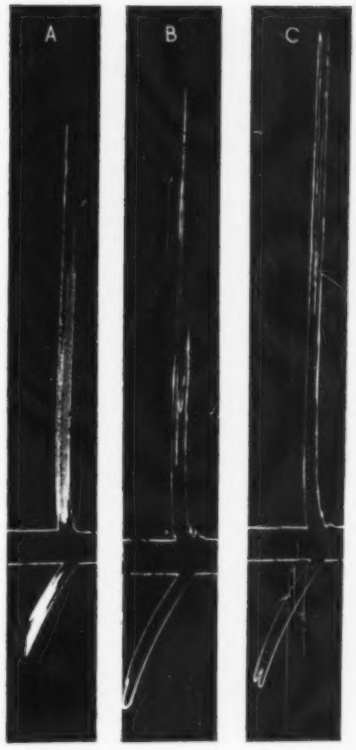

Fig. 10. Peristalsis in the guinea pig ileum recorded by Trendelenberg's method[54]. *Upper record:* contractions of longitudinal muscle layers. *Lower record:* peristalsis. Bath volume 70 ml. A. Normal record. B. Hexamethonium 1 mg. C. Tubocurarine 1 mg.

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Recent British Standards

Methods of Testing Vulcanized Rubber

This revised method (B.S.903:1960, price 4s. 6d.) replaces Part 23 of the 1950 edition of B.S.903—a standard which is in course of revision and issued in separate parts.

Apart from an alteration in the title, the main difference lies in the layout. The standard presents a full description of the various apparatus which may be used, together with details of the procedure to be followed and the method of expressing results. There are three full-page diagrams.

Copies of these Standards may be obtained from The British Standards Institution, Sales Branch, 2 Park Street, London, W.1.

COMPLEXOMETRIC ANALYSIS

by J. A. R. Genge, M.Sc., A.R.I.C.

Battersea College of Technology

PART III.—THE ANALYSIS OF METALS IN MIXTURES

QUITE apart from the various difficulties encountered in the complexometric analysis of individual metals and discussed in Part II, there is the additional problem that E.D.T.A. is not selective and, under normal circumstances, it is not possible to titrate for one metal in the presence of others. Various methods have been devised for overcoming this lack of selectivity, the method depending upon the components of the mixture, and the various methods will be surveyed to show how each may be applied.

The stability of a metal-E.D.T.A. complex increases with an increase in pH and, at any given pH, increases with an increase in the valancy of the metal. Also, since stability depends upon such factors as the polarizing power and the size of the ion, there will be considerable variation among metals of the same valency. Advantage may be taken of this, and Ringbom (1954) and Nogami and Nakagowa (1955) have determined the minimum and optimum pH values for complex formation between E.D.T.A. and a number of cations.

As an example of this, in Experiment 1, details are given for the separate analyses of magnesium and zinc in a mixture using eriochrome black T indicator. Zinc may be titrated at pH 6·8 without interference from the magnesium. After this, the pH can be readjusted to 10 and the magnesium titrated thus making the complete analysis a two stage direct titration.

Experiment 1

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.

Eriochrome black T solution—0·1 g. of the solid indicator dissolved in 100 ml. water made alkaline with a few drops of ammonia.

Buffer solution for pH 6·8—1·2 g. maleic acid dissolved in 20 ml. N sodium hydroxide and made up to 100 ml. with water.

Buffer solution for pH 10—7 g. ammonium chloride dissolved in 57 ml. 0·880 ammonia and made up to 100 ml. with water.

Procedure

An aliquot of the zinc-magnesium solution is neutralized to pH 7 with dilute ammonia using a minimum of universal indicator. Ten ml. of the maleate buffer is added, followed by a few drops of the eriochrome indicator, and the solution titrated with the standard E.D.T.A. until the colour changes from wine red to blue or grey. Five ml. of the ammonium chloride buffer is then added and a few more drops of indicator, if necessary, when the solution should again have a wine red colour. The second titration for magnesium is then carried out until the colour changes back to blue. Both end-points should be sharp.

Although M/100 is quoted for the strength of the E.D.T.A. and the metal solutions, the experiment may be carried out successfully at higher concentrations but it is generally true that end-points are better with dilute solutions. It is worth noting that, in this case, both analyses are carried out on the same aliquot of solution and with the same indicator. An alternative method for these two metals would be the titration of zinc alone at pH 4·5 (dithizone indicator) and, using a second aliquot, the titration of both metals at pH 10 (eriochrome indicator).

An example of this technique using two indicators is the determination of calcium and magnesium in the same solution and hence the estimation of water hardness. Details of this are given in Experiment 2. The method is based on the fact that while both metals can be determined with eriochrome, only calcium can be titrated with murexide indicator.

Experiment 2

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.

Eriochrome black T solution—as for Experiment 1.

Murexide solution—0·1 g. of the solid indicator (ammonium purpurate) dissolved in water.

Buffer solution for pH 10—as for Experiment 1.

Procedure

An aliquot of the calcium-magnesium solution is adjusted to pH 10 with dilute nitric acid or ammonia using methyl red indicator. Five ml. of the buffer solution and a few drops of the eriochrome indicator are added and the total metal content titrated with the E.D.T.A., the colour changing from wine red to blue at the end-point. Five ml. of 2 N sodium hydroxide and a few drops of murexide solution are added to a second aliquot and the calcium alone titrated with the E.D.T.A., the end-point in this case being from pink to violet. The magnesium can be obtained by difference.

It is necessary to use sodium hydroxide and not ammonia for the second titration as a pH of 12 or higher is required. Murexide is less stable than eriochrome and aqueous solutions should not be stored. If many of these titrations are contemplated, it is wiser to use both indicators as solids diluted with sodium chloride. For water hardness, only the first total titration with erio-

chrome indicator is necessary, the total hardness being expressed in terms of calcium. It should be noted that, if there is not much calcium in the water, a partial replacement titration with magnesium as described in Part II will be necessary to obtain a good end-point.

It is only in a limited number of cases that the differences in the stabilities of the metal-E.D.T.A. complexes are great enough, even at different pH values, to enable individual components of a mixture to be analysed. If however a reagent is available which will form a very stable complex with one component but has no effect on the other, then this can be used to prevent the first component from taking part in any titration. It is necessary that the complex formed be more stable than the corresponding E.D.T.A. complex with the metal. This technique is known as 'masking' and has been used very extensively. Perhaps the most versatile masking agent is the cyanide ion which forms stable complexes with cadmium, cobalt, copper, mercury, nickel, platinum metals and zinc and, as discussed by Flashka and Huditz (1952), enables mixtures including any one of these to be analysed. Other masking agents are triethanolamine for aluminium, indium and manganese and 2:3 dimercaptopropanol (B.A.L.) for a number of other metals. These are described by Pribil (1953 and 1954). There are many more including some which are specific for one metal as, for example, the iodide ion which forms the very stable HgI_4^{2-} ion with divalent mercury. Details are given in Experiment 3 for the determination of lead and zinc in the presence of each other by masking the zinc with cyanide.

Experiment 3

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.
Standard magnesium solution—approximately 0·01 M.
Eriochrome black T solution—as for Experiment 1.
Buffer solution for pH 10—as for Experiment 1.
5 per cent potassium cyanide solution.
Tartaric acid.

Procedure

About 1 g. of tartaric acid is added to an aliquot of the lead-zinc solution and the pH is adjusted to 10 with dilute ammonia. At this pH the lead would precipitate and the purpose of the tartaric acid is to keep the lead in solution as the tartrate complex which is, however, not strong enough to interfere with the E.D.T.A. titration. Five ml. of the buffer solution is added and the total metal content titrated with the E.D.T.A. Eriochrome indicator is used, changing from wine red to blue at the end-point. Five ml. of potassium cyanide solution is then added when the zinc cyanide complex is formed and the equivalent amount of E.D.T.A. is released. This is titrated with the standard magnesium solution until the indicator colour changes back to wine red.

The masking agent may be used directly or indirectly; thus it can be added before the titration or after a total titration as described in the example when an equivalent amount of E.D.T.A. is released. The former procedure has the advantage that only one standard solution (E.D.T.A.) is needed but two aliquots of the metal solution are required, one for the total metal determination and one for the separate titration of the unmasked metal. Usually this is the better method if two indicators are used but an indirect titration is convenient when analysing weighed samples of alloys.

The cyanide complexes of zinc and cadmium are relatively weak compared with those of other metals and if formaldehyde or chloral hydrate is added, cyanhydrin formation removes free cyanide ions from the solution and results in breakdown of the zinc or cadmium cyanide complexes. This process of 'demasking' can be used to analyse zinc or cadmium in the presence of other ions which also form cyanide complexes. Experiment 4 describes the analysis of nickel, zinc and magnesium in the presence of each other and use is made of both masking and demasking techniques.

Experiment 4

Reagents

Standard E.D.T.A. solution—approximately 0·01 M.
Standard magnesium solution—approximately 0·01 M.
Eriochrome black T solution—as for Experiment 1.
Buffer solution for pH 10—as for Experiment 1.
5 per cent potassium cyanide solution.
5 per cent formaldehyde solution.

Procedure

An excess of E.D.T.A. (at least twice that required to complex all the metals present) is added to an aliquot of the metal solution and the pH is adjusted to 10 with dilute ammonia using methyl red indicator. Five ml. of buffer solution and a few drops of erichrome are added and the excess E.D.T.A. back titrated with magnesium giving a value for the total metals present. Ten ml. of potassium cyanide solution is then added to complex the nickel and zinc and the equivalent amount of E.D.T.A. liberated is titrated with the magnesium. Finally 5 ml. of formaldehyde solution is added to demask the zinc which is titrated directly with the standard E.D.T.A.

It is advisable to avoid a large excess of cyanide or formaldehyde solutions as these tend to affect the end-point. Usually it is necessary to readjust the pH and to add a little more indicator before the final titration.

These two examples will give some idea of the versatility of the masking technique. In theory it is possible to add more than one masking agent and titrate at different pH values with different indicators and so analyse almost any mixture. In practice the end-point worsens as more materials are added to the solution. In addition there is the difficulty that all materials must be pure.

Although the above techniques are very useful, they can be applied only to a limited number of mixtures and in the majority of cases, some preliminary separation is necessary. Some very elegant separations have been developed, such as ion exchange, although this is more often useful for separating unwanted anions. Zinc may be determined by solvent extraction using excess potassium thiocyanate and shaking with methyl isobutyl ketone. Similarly a number of metals can be extracted with a solution of sodium diethyldithiocarbamate in carbon tetrachloride or chloroform. This extraction is limited to lead and bismuth if carried out on an ammoniacal cyanide solution and use is made of this fact in Experiment 5 which describes the procedure for analysing a mixture of lead and zinc.

Experiment 5

Reagents

Standard E.D.T.A. solution – approximately 0·01 M.
Eriochrome black T solution – as for Experiment 1.
Buffer solution for pH 10 – as for Experiment 1.
2 per cent solution of sodium diethyldithiocarbamate in water.
10 per cent potassium cyanide solution.
Tartaric acid.
Chloroform.

Procedure

About 1 g. of tartaric acid is added to an aliquot of the lead-zinc solution to prevent the lead from precipitating and the solution adjusted to pH 10 in the usual way. Five ml. buffer solution and a little eriochrome indicator are added and the total metals titrated with E.D.T.A. solution, the colour change being wine red to blue. One g. tartaric acid, 50 ml. potassium cyanide solution, excess ammonia and 10 ml. of the sodium diethyldithiocarbamate solution are added to a second aliquot which is then shaken in a separating funnel with one 10 ml. and two 5 m. portions of chloroform. The combined chloroform extracts are mixed with 10 ml. dilute nitric acid and boiled gently until all the solvent has been evaporated. A further 1 g. tartaric acid is added, the pH adjusted to 10, buffer solution and eriochrome indicator added and the lead titrated with E.D.T.A.

As shown by Kinnunen and Wennerstrand (1954), techniques of this sort can be very useful for analysing alloys and ores when normal chemical separations are often very tedious. In a large number of cases, however, there is no satisfactory alternative to a quantitative precipitation. Thus many metals can be separated easily as the sulphide and, in fact, most of the well established precipitating agents may be used in conjunction with an E.D.T.A. titration. For example, sulphate can be used for barium, dimethylglyoxime for nickel, oxalate for the rare earths and 8-hydroxyquinoline for aluminium and a number of other metals.

It will be appreciated that E.D.T.A. has proved to be a very versatile reagent for analysing mixtures. The experiments described are examples of techniques and involve ideal mixtures of metals but the same methods can be applied to 'practical' problems such as the analyses of alloys, ores, plating solutions and trace quantities of metallic impurities. It is not always possible to carry out the complete analysis with E.D.T.A. but it is usually possible to analyse one or more components more easily by this method than by any other.

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Meldola Medal for 1960

The Meldola Medal is the gift of the Society of Maccabaeans and is normally awarded annually. The next award will be made early in 1961 to the chemist who, being a British subject and under 30 years of age at December 31, 1960, shows the most promise as indicated by his or her published chemical work brought to the notice of the Council of the Royal Institute of Chemistry before December 31, 1960.

No restrictions are placed upon the kind of chemical work or the place in which it is conducted. The merits of the work may be brought to the notice of the Council, either by persons who desire to recommend the candidate or by the candidate himself, by letter addressed to The President, The Royal Institute of Chemistry, 30 Russell Square, London, W.C.1, the envelope being marked 'Meldola Medal'.

The letter should be accompanied by six copies of a short statement on the candidate's career (date of birth, education and experience, degrees and other qualifications, special awards, etc., with dates) and of a list of titles, with references, of papers or other works published by the candidate, independently or jointly. Candidates are also advised to forward one reprint of each published paper of which copies are available.

Forthcoming Conferences

A Conference on Optical Instruments and Techniques is being arranged by the British National Committee for Physics, under the auspices of the International Commission for Optics. It will be held in London during the period July 10 to 14, 1961. Enquiries should be addressed to Mr. K. J. Habell, National Physical Laboratory, Teddington, Middlesex, England.

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The Institute of Physics and the Physical Society announces that it is arranging a conference on statistical mechanics (with special reference to irreversibility). It will be held at Queen Mary College, London, on Monday and Tuesday, 19 and 20 December, 1960.

A full programme has already been arranged. The topics include the general theory of irreversibility, random processes, liquids (theory and experiment) and irreversibility in gases and in plasma.

Further information and preliminary programmes can be obtained from the Secretary, The Institute of Physics and The Physical Society, 47 Belgrave Square, London, S.W.1.

ARTICLES

THEORY AND PRACTICE IN THE PHYSICAL CHEMISTRY LABORATORY

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PART VIII—SOME ASPECTS OF EVALUATION OF pH VALUES

THE determination of pH values is of fundamental importance not only in the purely academic fields, but also industrially, biochemically and medically. Sufficient variation in the pH values affects many chemical reactions and processes to such an extent as to result in reversal of the original reaction.

Degrees of acidity and alkalinity have been appreciated for many years; thus the dissolving effect of vinegar on teeth, and the use of (alkaline) soaps have been quoted as early as Biblical times. However, as in the case of many other '*capacity*' factors, understanding of the effects of hydrogen ions or hydroxyl ions on various processes could not be accurately or easily evaluated until the quantitative concept of pH and convenient methods for its accurate measurement were well developed.

The theoretical concept of hydrogen ions was first mooted as a result of the process of electrolysis of acid solutions, when it was shown that the liberation of hydrogen gas was explainable in terms of the dissociation of the parent acid molecule to give a positively charged H^+ which travelled towards the cathode and a negatively charged anion which travelled towards the anode. The concept that $H^+ + OH^- \rightleftharpoons H_2O$, and the Law of Mass Action application which resulted in this equilibrium being formulated as $[H^+][OH^-] = K_w$, allowed correlation between the concept of alkalinity in the field of hydroxyl ion preponderance and the concept of acidity with hydrogen ion preponderance. The vast range of $[H^+]$ variation from approximately unity in N HCl to approximately 10^{-14} in N KOH solution made it inconvenient, if not difficult, to handle the various values encountered.

It is to Sörensen that we owe the definition of pH, which concept has considerably simplified consideration of problems involving hydrogen ion concentration. Sörensen advanced the definition

$$pH = -\log [H^+]$$

and this pH scale is now used extensively.

It is essential to remember that the pH scale is a logarithmic scale and not a simple numerical one. Thus, in going from pH 1 to pH 2 the hydrogen ion concentration changes from $[H^+] = 1$ to $[H^+] = 0{\cdot}1$; in going from pH 2 to pH 3 the $[H^+]$ changes from $[H^+] = 0{\cdot}1$ to $[H^+] = 0{\cdot}01$. Putting this in terms of a mathematical equation we have for the difference in pH values (dpH) of two hydrogen ion concentrations $[H^+]_1$ and $[H^+]_2$

$$dpH = pH_1 - pH_2 = -\log [H^+]_2/[H^+]_1$$

This concept is generally understood, but the associated factor of variable buffering capacity in different pH ranges is sometimes forgotten.

The above definition, while used extensively, cannot be considered sufficiently accurate in fields where ionic activities play a decisive role. The modern definition of $pH = -\log a_{H^+}$ then represents the accurate standard of pH values and should be used instead of the original Sörensen definition.

It must be emphasized that the modern and most common instrument of pH measurement, viz. the pH meter employing a glass electrode system, is calibrated in terms of the modern definition. Thus, at 20°C. when one uses a 0·05 M. potassium hydrogen phthalate solution to standardize the glass electrode at pH 4·00 and 0·05 M. borate solution to agree at pH 9·18,* the scale employed involves

$$pH_{phthalate} = 4{\cdot}00 = -\log \{ [H^+] \times f_{H^+} \}_{phthalate}$$

and

$$pH_{borate} = 9{\cdot}18 = -\log \{ [H^+] \times f_{H^+} \}_{borate}$$

where f_{H^+} is taken to represent the activity coefficient of the hydrogen ion, but is *experimentally* a *geometric mean* activity coefficient of the respective ions of the particular electrolyte. Thus in the case of the borate solution, it would be more accurate to write for the above respective conditions

$$4{\cdot}00 = -\log \{ [H^+] \times f_{\pm} \}_{phthalate}$$

and

$$9{\cdot}18 = -\log \{ [H^+] \times f_{\pm} \}_{borate}$$

The need to employ the geometric mean activity coefficients ($f_\pm$) instead of the specific ones results from the experimental limitations.**

All accurate standardizations, including the above calibrations, are based on values established using the hydrogen electrode and the relationship

$$E_{H_2} = \frac{-RT \ln[H^+]}{F} = +\frac{2{\cdot}3026\ RT\ pH}{F}$$

*For references *see* Lewin (1955).

**For a discussion of activity coefficients *see* Lewin (1960).

where R = 8·3144 volt-coulombs/(degree mole)
F = 96,496 coulombs
and T = the absolute temperature.

In order to determine the pH of the given test solution one should determine the e.m.f. of the hydrogen electrode in presence of the solution and calculate the pH value of the above equation. This can be done in the laboratory by combining the hydrogen electrode with a suitable reference electrode such as the calomel or the silver-silver chloride electrode. The procedure involves the initial potentiometric evaluation of the cell e.m.f. As the e.m.f. of the cell is equal to the *sum* of the e.m.f.s. of the two half-cells, calculations using the *correct* sign of the e.m.f. of the co-electrode gives the required E_{H_2}.

Now, while the reference electrode is usually a convenient and stable electrode, the hydrogen electrode is experimentally somewhat temperamental. The platinum black deposit, which forms the catalytic surface on the hydrogen electrode where the dissolved hydrogen undergoes the reversible reaction

$$H_2 \rightleftharpoons 2H^+ + 2\ominus$$

is easily poisoned or inactivated. This inactivation can be noted in the associated instability of the cell potential. Thus, when the oxygen free hydrogen is passed through the hydrogen electrode, the value of the cell potential rises as the solution becomes deoxygenated, resulting after about five to ten minutes in a stable potential. Occasionally, however, and this tends to take place more often when the hydrogen has passed through the electrode for some time, the cell e.m.f. remains stable for only a very short period or not at all. We have noted repeatedly that, when this happens, removing the platinum electrode, dipping it into conc. nitric acid for a few seconds, subsequent thorough washing with distilled water and the test solution and replacement in the electrode compartment results in very stable potentials being obtained. Other instabilities or very slow potential drifts can be traced to other causes. Sometimes these may be ascribed to diffusion of KCl from the saturated KCl bridge.

It is common practice to use saturated KCl agar bridges to eliminate liquid junction potentials. The principle is sound, but the *duration* of the experiment can result in a significant error.

It is usually assumed that KCl diffusion from the extremely minute agar capillaries is negligible when a small bore glass tube – c. 0·2 cm. in diameter – is employed. This assumption is correct when the duration of contact is short, e.g. five to ten minutes. However, when longer experimental contact periods are encountered this does not hold. We have carried out a number of experiments to determine the effect of contact between the saturated agar KCl bridge and the test solutions, which were KCl free, on the accuracy and reproducibility of the recorded e.m.f. values. Even at low rate of hydrogen bubbling through the test solution, a difference of as much as five millivolts could be observed after an hour's run, when potentiometers capable of 0·2 millivolt accuracy were used.

The diffusion across the liquid junction between the saturated KCl solution and the test solution is, of course two directional, viz. of the test solution into the space occupied by the bridge KCl, and of the KCl from the bridge into the test solution. The diffusion of KCl into the test solution can be readily shown in control tests for the chloride ion. The diffusion of the test solution in the opposite direction can be demonstrated clearly using a coloured test solution such as that containing methylene blue. The methylene blue can be seen to creep slowly up the agar gel, the creep amounting to as much as 0·3 cm. after an hour's contact.

It is possible to limit the errors due to diffusion by the use of flowing liquid junction and by restriction of the area of contact between the KCl bridge and the test solution. The use of the first method is however often inconvenient – particularly when only a small quantity of test solution is available. The latter method is associated with considerable impedance which results in the need for amplifying systems such as that found in a valve potentiometer.

The diffusion of the test solution *into* the bridge can be eliminated if one dispenses with the agar medium as a semi-solid supporter for the KCl solution. The agar is generally used as a supporting medium which stops any syphoning effects. As such it fulfils its function quite well, but by its very nature it does not allow offsetting diffusion of the test solution into the bridge by means of a counter-current of the KCl solution. If we dispense with the agar as a supporting medium for the bridge and use instead the KCl solution in a glass tube the ends of which can be blocked by sintered glass, ground glass joints, or asbestos the KCl diffusion can be considerably decreased. Nevertheless, it is possible to ensure a slight flow of KCl solution past the block on application of hydrostatic pressure. The slight flow, which may be varied from one drop per six hours to one drop per minute, can be regulated to overcome the creep of the test solution into the bridge. The 'contamination' of the test solution by the KCl can be greatly minimized by a 'flowing junction' type of contact allowing external drainage or by using a comparatively large volume of test solution. The 'contamination' is readily calculable and can be arranged to be as small as permissible. Thus, suppose that one drop of KCl is lost per 200 minutes. Let the experimental measurement duration be 20 minutes, then 1/10th of a drop enters the test solution in the 20 minutes and it is equivalent to 1/200 ml. (as one drop equals 1/20 ml.). This quantity is dissolved in, say, 50 ml. of test solution. The final concentration of the KCl in the test solution is approximately

$$\frac{(1/200) \times (\approx 4)}{\times 50} \approx 4 \times 10^{-4}\ \text{N KCl}$$

(assuming the normality of the KCl solution to be roughly 4N.)

The effect of this small concentration of the neutral KCl on the pH of the test solution depends on the ionic strength of the test solution itself. The more dilute the solution is the smaller is its initial ionic strength and

the greater would be the effect of the KCl introduced. However, the solution will have to be very dilute for such a concentration of KCl to have any effect. Consider a 0·01 N HCl solution which has as ionic strength of 0·01. The introduction of the above quantity of KCl increases its ionic strength by about 4 per cent and may alter the pH only slightly, viz. in one or two places in the *third* decimal. In other cases, where the initial ionic strength of the test solution is higher, the effect on the pH value is even smaller. The KCl diffusion effect can be decreased still further by decreasing the rate of KCl seepage or by increasing the volume of the test solution. Further, a pH value correct to the third decimal is rarely required and invariably involves the problem as to its precise evaluation, because it is not certain whether a saturated KCl bridge eliminates completely the liquid junction potential as far as the third decimal point is concerned.

For the above reasons the use of the above restrictive junction of KCl solution bridge is preferable to the use of an agar bridge. Unfortunately, as the flow is restricted the electrical resistance across the flow restriction unit increases and may well reach a value which results in experimental uncertainty in several millivolts. This difficulty can be overcome by the use of amplifying systems such as that of a valve potentiometer.

Valve potentiometers are used extensively in glass electrode pH determinations in research laboratories where their principles are sufficiently understood, or in industrial laboratories where the operations are routine and where the operator does not have to appreciate the scientific principles involved*. However, the use of a valve potentiometer by a student who has not previously encountered it calls for some consideration. When a trained worker uses an ordinary potentiometer for the determination of the e.m.f. of the hydrogen electrode, he should understand the electrical circuits as he had been taught the workings of a potentiometer. The basic principles are so elementary that there need be no fear that he will carry out the e.m.f. determinations without understanding what he does even when he uses a closed box dial potentiometer instead of the ordinary bridge type first encountered. However, the valve potentiometer involves principles which the student is often unlikely to have encountered. Should students use instruments with the principles of which they are unfamiliar? From the strictly educational point of view the answer must be a firm negative. If students are to be allowed to operate instruments the workings of which they do not understand, they will get accustomed to the notion that they need not understand the workings of scientific instruments that they are to use. Once imbued with this idea they will tend to extend it further and apply it to their studies in that they will tend to learn parrot-fashion scientific facts and figures without attempting to understand their background and meaning and without trying to identify the scientific patterns concerned. It is essential to discourage such tendencies and to emphasize to the students that one of the first principles in science is not to take anything for granted. In this connection it is convenient to distinguish between the period when scientific education is taking place, when every effort should be made to encourage the students to develop their logical approach to various problems, and subsequent periods when compromises between ideality and practice are such as to appear to negate educational principles. The cynic would argue that in this case scientific education might well be considered wasteful, but this depends on what one means by the term 'education'. Education has been defined in various ways, and one of the most outstanding definitions is: Education is that which a person has retained when he has forgotten the other things he has been taught during his school and college days. On this basis scientific education is best defined as the imbuing of a scientific *attitude of mind* rather than that of acquiring of scientific knowledge.

Returning to the introduction of the use of valve potentiometers, a student proceeding to carry out pH evaluations by means of the hydrogen electrode should use at first the normal potentiometric arrangement. Thereafter, having encountered examples involving high electrical resistance and after tutorials in which ways of overcoming such difficulties have been dealt with, the principles and circuits involved in the valve potentiometer should be explained to him. He should *not* be allowed merely to exchange the ordinary potentiometer for the valve potentiometer and then proceed to take measurements. The above system in which a student is allowed to use a valve potentiometer after a comparatively short explanation is not really satisfactory as it represents a compromise between a full course in electronic principles for chemistry students and no course at all. The lack of any electronic course for chemistry students arises from their non-inclusion in several chemistry degree, Grad.R.I.C., H.N.C. and parallel qualifications. It is indeed surprising that in this age of electronic instrumentation not even a short course of electronics is insisted on by the various examination bodies concerned. Instead, considerable time is devoted to some old methods and techniques. There is no doubt that in the future this attitude will change. But in the meantime short electronic instrumentation courses should be included in chemistry courses as a compulsory and not an optional subject, because there are many students who interpret the function of teaching institutions to be that of transferring to them only that scientific information which forms part of the syllabus. Everything else is considered almost 'taboo', because of lack of time. It is the function of the teaching institution to define the education to be given, and we believe that one of their major functions is to ensure that their graduates are not 'uneducated scientists'. In this connection the laboratory

(*Concluded on page 724*)

*For example, an employee operating a controlling system for adding acid or alkali to a chemical process unit, which has to be at constant pH value, need not understand the workings of the pH meter or automatic pH recorder in order to keep the routine process in continuous operation.

FOOD SCIENCE SECTION

THE DEVELOPMENT OF FOOD AND DRUGS LAW IN ENGLAND

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PART II

From 1930 to 1940

The 1938 Act[11].—The Food and Drugs Act, 1938 incorporated for the first time in one statute food and drugs legislation with public health legislation relating to foodstuffs: the dichotomy of practice and purpose, of course, remained. Important changes included penalties for false or misleading labels and advertisements of food or drugs, and power to make regulations regarding the preparation, storage, sale and delivery of foods. Where an officer procured a sample which did not need analysis, he was relieved from the duty of submitting it to a Public Analyst. A Departmental Committee on the Composition and Description of Food (in 1934) had recommended that the law be so altered.

Administrative Action, 1930-1940.—The National percentage of adulterated samples in 1938 was 5·7[12]. Infringements of the Preservatives Regulations still occurred and adulterations of milk were still reported. Cream, cheese, butter, margarine, lard, vinegar, spirits and suet were reported on a relatively small scale. Infringements relating to drugs were still being found. Two particular developments in control during the decade 1930 to 1940 should be mentioned. Adulteration in the sense of sophistication of the substance of foods and drugs, whether natural or compounded, appears to have been substantially controlled, but the prohibition against selling an article of food not of the nature demanded by the purchaser had not been particularly invoked. About 1933 the Middlesex County Council took the initiative in discovery of cases where different, usually inferior, varieties of natural foods were passed-off for some other variety and a large number of prosecutions ensued. With regard to fish, for example, it was found that witch, megrim or lemon sole were passed-off as 'Sole' or 'Dover Sole'; haddock, saithe and pollock were sold as 'Hake'; whiting as 'Haddock'; smoked whiting and smoked cod as 'Smoked Haddock'; dabs and flounders as 'Plaice'; ling as 'Cod'; brill as 'Turbot'; pilchards and large sprats as 'Herrings'; various varieties of plums may be passed-off as 'Victoria Plums' and apples as 'Cox's Orange Pippin'. Often allied to these mis-descriptions, verbally given at the time of sale, were false labels applied to the goods. Before the 1938 Act recourse was made to the Merchandise Marks Act, 1887, section 2 of which prohibits written false descriptions being applied to goods. The same Act is still used to supplement deficiencies in other controls, for example, the misdescription of the country of origin of foodstuffs made by label; thus in 1952 to 1959 the Middlesex County Council instituted 263 prosecutions for that offence, 205 of them relating to meat. Secondly, after 1930 enforcement of the Merchandise Marks Act, 1926 and the Imported Food Orders commenced. This duty, permissive and not mandatory, is to ensure that imported food such as meat, fresh apples, raw tomatoes, eggs in shell, dead poultry, butter, bacon and ham, are marked with their origin (the word 'Foreign' or 'Empire', as appropriate, or the actual name of the country), thus protecting both the consumer and the home-producer. In seven complete years before 1939, in Middlesex alone, 650 summonses and 741 cautions were issued for infringements of the Orders. The worst trouble was found in respect of meat (307:127), apples (146:287), tomatoes (107:167), butter (45:82) and poultry (36:16).

The Position in 1960

The Food and Drugs Act, 1955[13].—The present law is enshrined in the Food and Drugs Act, 1955. This Act incorporated the important provisions of the Act of 1938 together with a great deal of emergency legislation arising from war-time control, which developed the pattern of food and drugs administration. From this very short article details must be excluded. During the war it was necessary strictly to control foodstuffs as to composition, price and marking. In 1943 the Defence (Sale of Food) Regulations temporarily replaced and extended the labelling and advertisement provisions in relation to food of the Food and Drugs Act, 1938. Power was given to make regulations; firstly with minimum standards of composition for foodstuffs, and secondly requiring them to be labelled. Both food standards orders and pre-packed food labelling requirements (descriptions, ingredients, packer's name and address, etc.) were evolved from these and they were preserved by the 1955 Act. Regulations for safe milk requiring sale under special designations which had been further developed during the 1940's were also incorporated. Special provisions protecting the use of the word 'cream' whether sold as a simple article or as an ingredient were introduced. Broadly, the 1955 Act in the present state of the law appears as the fruition

into a sound instrument of the control commenced one hundred years ago. The Act itself contains the broad and simple prohibitions particularly initiated in 1875. For example, the preparation and sale of injurious foods and adulterated drugs (s.1); the general protection to purchasers of food and drugs sold not of the nature, substance or quality they are deemed to expect (s.2); the prohibition of false or misleading labelling and advertising of a food or drugs (s.6); the punishment for the sale of food which is intended for, but unfit for, human consumption (s.8) and with regard to specific foods, for example, the requirement that in specified areas only safe 'Tuberculin Tested', 'Pasteurized' or 'Sterilized' milk be sold by retail. To meet the revolutionary growth of compounded foods and the distribution of pre-packed articles in huge quantities, a fluid control of regulation-making powers is provided. Thus for example s.4 gives wide powers to make regulations as to the composition of food, and as a result a number of foods has been subjected to standards orders (e.g. the control of water in butter and margarine formerly prescribed by statute; standards for preserves; fish cakes; meat cakes; ice cream; cream, and so on), and prescriptions of authorized substances in food have been made. S.7 gives wide powers to make regulations as to the labelling and description of food, and the Labelling of Food Order, 1953 comes under this heading. In 1947, an Advisory Committee, called the Food Standards Committee, was set up and now advises the Ministers concerned on the composition, description, labelling and advertising of food. It has issued many reports of great interest, notably in connection with preservatives in food, and in supplement to the Preservatives Regulations, as they have been amended, colouring matters in food are now controlled by the prohibition of all but known safe substances. So far as labelling is concerned various labelling requirements have been made, but there is slow development in the specific requirements as to advertisements of foodstuffs. The main prohibitions of the Act and Order and the Regulations are still absolute, but since 1938 it has been possible by way of defence for a defendant to escape penalty in most cases by bringing before the Court another person to whom he alleges the contravention was due, and if the first defendant proves that to be so, and that he used all due diligence, he may be acquitted. A prosecuting authority, if satisfied that the offence was due to the act or default of some person other than the prime offender, may by-pass the latter person and prosecute the former for the offence. The harsh and rigorous application of the concept of absolute liability, which was strong in law in 1875, has to some extent been relaxed in recent years.

Administration in 1960.—Unfortunately, for many years there have been no National statistics of food and drugs enforcement as formerly were to be found annually in the reports of the Local Government Board. There is little doubt, however, that the problems which the modern administrator faces are different from those which pertained even 20 years ago[14]. Offences due to the food retailer largely fall within the area of passing-off of natural foods. The great extent of pre-packing and compounding of foodstuffs and distributing them over wide areas, the growth of specific requirements as to composition and declaration of contents by label, the removal to a large extent of the possibility of gain by the local trader in adulterating or sophisticating the wares he sells, have resulted in a pretty widespread abolition of the use of adulterants. There remains a constant need to ensure that there is no passing-off of inferior for superior natural foods by retailers, nor mismarking of them. So far as the sellers of pre-packed compounded foods are concerned, control and argument turn more to what shall be regarded as a fair minimum composition of compounded articles. Generally, this does not lead to requesting a Court to decide, as it has power to do when a prosecution is brought under the Act, for there has developed a tendency to seek by negotiation agreement between the trade and officials. Large manufacturing and marketing firms distributing pre-packed goods advertise extensively, and of course produce labels on all their wares. There is constant need to keep control of statements and claims in labels and advertisements, and immediately after the war the Ministry of Food (as it then was) was active and produced a useful book[15] laying down certain rules with regard to claims in labels and advertisements, and incorporating a number of Codes of Practice agreed between the Ministry and the trade as to the composition and labelling of certain foodstuffs. This device the Ministry discontinued about 1955 as it did its advisory service on labelling, which was available to persons who requested it. The discontinuance of this work caused it to be taken up by a number of the leading enforcing authorities, and some of them have a great deal of requests for advice from traders, advertising agents and the like. There has recently been set up a Joint Advisory Committee consisting of the Enforcing Authority Associations, with Public Analyst representation, to pick up and continue negotiation and revision of Codes of Practice with the trade. Effective enforcement is a matter of expertise and it is the recognition of this which has been slow in developing. The function of the food and drugs administrator is to be master of practical enforcement in his own area, responsible only to his Local Authority and supported by the advice insofar as analysis is concerned of his Public Analyst[16]. His practical task is to maintain his knowledge of the instruments which are to hand, to create new ones, to enforce and to control by policy-making the work of his sampling officers. All effectiveness depends on the exercise of choice – choice of samples, choice of technique, choice of enforcing instrument. Food and drugs administration involves the knowledge of the whole art and technique of enforcement of prohibitive legislation backed with penal sanctions: knowledge of the law, traders, trade practices, the nature and composition of foodstuffs and drugs, dietetics, nutrition and the understanding and expectation of the public. All this and more is the amalgam which the enforcing authority alone can combine and assess in its decisions. There is no illusion

that has been more persistent than that Food and Drugs administration is a Public Health matter. It is nothing of the sort. It is aimed primarily at protecting the ordinary consumers' desire and the honest traders' standards. It is a constant battle against the debasement of the coinage of nomenclature and deterimental variations and substitutions in food composition.

Conclusion

It is concluded that in its structure the present law has developed soundly. There are comprehensive and simple prohibitions in themselves sufficient to deal with all foreseeable instances of adulteration or passing-off, and with false or misleading labels or advertisements. There are wide powers available prudently and expeditiously to control the composition and labelling and advertising of specific foodstuffs; there are sound advisory bodies, for example, the Food Standards Committee and the Food Hygiene Advisory Council and for preliminary standards or codes of practice, the new Joint Advisory Committee of local authorities.

The Local Authorities.—We suggest that there is one cardinal error left in the structure. There has always been a large number of local authorities entrusted with enforcement. In 1875, and for many years afterwards, there were a number with little more than a population of 10,000. For many years the total number of authorities was in the nature of 230 to 240; the present number is about 280. The Act of 1955 provides generally for authorities of 40,000 to become Food and Drugs Authorities and exceptionally for those with as low a population as 20,000. The development of efficient administration, particularly in smaller districts, was slow. Thus in 1905[6] the Local Government Board expressed concern at an often ineffective state of affairs, and said that common faults were that there was no senior direction of the work, that the sampling officer rarely had special aptitude or training, that the number of samples taken was insufficient to encourage him to study methods calculated to detect practices of adulteration and as a rule he was well-known to traders in the district, that samples tended to be taken in batches, that detective methods by using agents were not employed and so forth. It is argued that the modern methods of distributing foods have created conditions which make it wholly desirable that the enforcing authorities should be only the largest available. Only the very largest authorities have resources adequate to pursue full scale investigations without financial embarrassment, and a population and area large enough to justify the employment of officers specializing in the enforcement of this legislation. To avoid expense and wasteful duplication, and to achieve optimum efficiency and creative drive there is overwhelming advantage in the aggregation of enforcement. Officers of large authorities avoid being the personal familiar of local traders but retain local knowledge; enforcement machinery to be effective and efficient must avoid multiplication of action by different authorities, and variable treatment of the same traders. Having regard to the increase in mobile trading fluid through small districts, to the distribution of pre-packed goods over wide areas, to the large trading areas of individual multiple shop firms and to the distribution of country-wide advertisements, the need that administration should be geared to the structure of distribution and marketing makes County Councils the ideal authority. The advantages of local authorities' control, however, remain. In the outcome they can best provide an alert, active and expert administration with results which are all the more powerful and sound for being on the one hand due to independent initiative, and on the other, pursued in concert with their colleagues.

Cosmetics.—The last comment is that the next major extension of the law might be the control of cosmetics. By analogy with the control of food, a range of goods of common consumption, being processed, compounded, pre-packed articles, characterized by chemical sophistication, liable to introduce health hazards to the user, and sold in the competitive market by heavy advertising, and in every variety of container and wrapper, and, moreover, being goods used to assuage basic human desires, *prima facie*, raises the need to be controlled in the interests of the consumer. Such control has been in existence in the United States of America and in Canada for some 20 years; both countries having seen fit to prohibit the use of coal-tar dyes and other toxic substances in cosmetics, and to control labelling and advertisements with regard thereto.

REFERENCES AND NOTES

[11] For the law in 1940, see R. A. Robinson and R. Ives (1939) 'Bell's Sale of Food and Drugs', 10th Edn.
[12] Sale of Food and Drugs: Reports of Public Analysts, 1938, Ministry of Health.
[13] For the law in 1960, see J. A. O'Keefe (1956) 'Bell's Sale of Food and Drugs', 13th Edn.
[14] J. A. O'Keefe (1953) 'Some Aspects of the Modern Range of Food and Drugs Work'; Institute of W. & M. Admin.
[15] Report of the Ministry of Food (1949) 'The Advertising, Labelling and Composition of Food'.
[16] Cf. the discussion at pp. 219-224, Monthly Review, Oct., 1959: Institute of W. & M. Admin.

Theory and Practice in the Physical Chemistry Laboratory. (*Concluded from page 721*)

period can be used correctly in the field of pH measurements. Not only the method and principles should be demonstrated, but the sources of error and the limitations should be considered in detail. The fact that valve potentiometers can overcome certain difficulties in pH measurements should be used to interest the students in electronic instrumentation. This could be further strengthened by demonstration of electronic pH and other recorders, thus implanting in the student's mind the need to study the subject and make it easier to accept the compulsion involved in attending such courses.

There are several other aspects of pH measurements which will be discussed in other communications.

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Lewin, S. (1955). Lab. Practice, **4**, 441.
Lewin, S. (1960). 'The Solubility Product Principle'. Pitman & Sons.

CLINICAL BIOCHEMISTRY

by E. J. King, D.Sc., Ph.D., F.R.I.C.

Professor of Biochemistry, British Postgraduate Medical School

PART II

Automatic Analytical Apparatus

Automatic Pipette.—An automatic pipette invented by Wootton (1957) uses the 'milking' principle of drawing a liquid along a tube by applying a moving pressure to the tube over a defined distance thereby drawing the liquid along the tube in the wake of the pressure. The instrument delivers 0·3 ml. of fluid, or multiples of 0·3 ml., at a high rate and with an accuracy of about one per cent. (Fig. 8).

Autoanalyser.—The Technicon Autoanalyser is the instrument of which we have had most experience. It is manufactured by the Technicon Instruments Company Limited and is also on display. The principle is simple, and is illustrated in Fig. 9. A turntable carries the samples, either neat or suitably diluted, and several standard solutions to cover the expected range of concentrations. These are sucked, each for a defined period, along a plastic tube, each sample being separated from the following one by a short column of air, which enters the tube as its sampling end lifts automatically out of one little plastic beaker in the turntable to dip into the next one as it turns into position. (Fig. 10). The samples, and standards, pass round and round through a long spiral groove, and back again, in a plastic plate against a dialysing membrane on the other side of which another fluid, containing one of the reagents for the analytical reaction, moves in a similar groove of a second plate which is the mirror image of the first. Into the reagent stream the diffusable constituents of the sample, or of the standard, pass to react with the reagent in the stream on the other side of the membrane (Fig. 11). When the two streams emerge from the plate, that of the exhausted samples or standards passes into the waste, while the reagent stream goes on to mix with converging streams of one or more further necessary reagents for the analytical reaction. It is important to notice, at this stage, that it is not essential for the success of the operation that all of the analysable constituent under investigation should dialyse from the sample or standard through the membrane into the reagent; provided only that the same proportion of the constituent pass into the reagent from both the sample and the standard, then the analysis will be successful and accurate. As several standards are always used in any particular analysis there will always be a final standard tracing to correspond nearly to any particular sample.

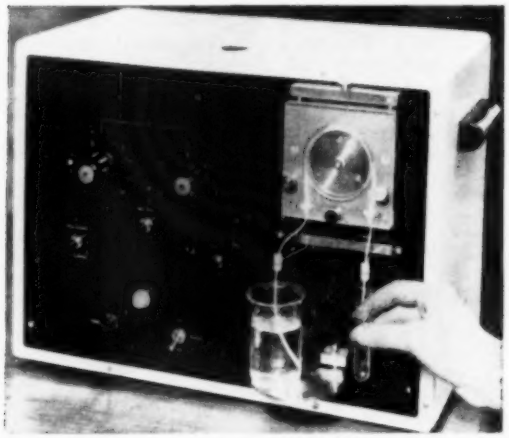


Fig. 8. Automatic pipette (Wootton, 1957). The instrument delivers 0·3 ml. of fluid or multiples of 0·3 ml. (Baird & Tatlock Ltd.)

The next stage for the reaction may require heat, and the column of liquid may therefore pass next through a cooker, whose temperature may be suitably adjusted to accomplish the desired reaction. The coloured substance so formed then passes into a cuvette which stands before a photo-electric cell. This is attached to a recording galvanometer, which writes the optical densities of different reacted samples and standards as they come to the photo-electric device. The result is a tracing consisting of maxima and minima, which correspond to the peaks of optical density for the different samples and standards and for the blank unreacted and uncoloured reagent between them. It is then a simple matter to read off the concentration of any sample from a standard whose peak corresponds with it or from two standards between whose peaks it lies. Inorganic substances commonly determined with the Autoanalyser are phosphate, chloride, carbon dioxide, calcium, and in fact any inorganic substance for which there is a chemical reaction which will lead to a coloured product.

Some Organic Analyses

Autoanalyser.—The same principle of using a chemical reaction to produce a coloured product is employed for the determination of organic substances with the Autoanalyser. Urea, uric acid, creatinine and sugar are commonly determined constituents of blood whose

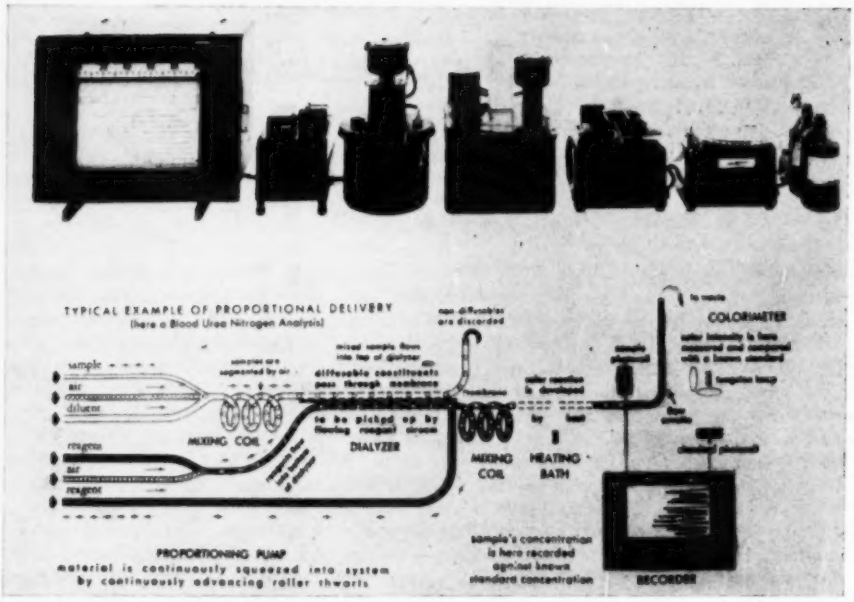


Fig. 9. The Technicon Autoanalyser. Apparatus and scheme of operation.

estimation is of diagnostic importance, and for which chemical reactions have long been used to produce coloured products which can be measured with a photoelectric photometer. The methods used with the Autoanalyser are in most instances the same as, or slight modifications of, existing methods of colorimetric analysis. Fig. 12 shows a galvanometer recording of a set of sugar standards of different concentrations prepared to match the expected results for a number of blood samples from normal persons and clinical patients. On the right of the figure are seen the peaks corresponding to the various standards; to the left of them are the peaks for the various samples of blood. The concentration of any specimen is easily read off by comparing its peak with that of a corresponding standard or with the one nearest to it.

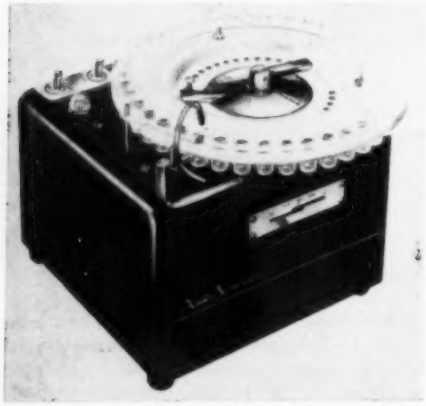
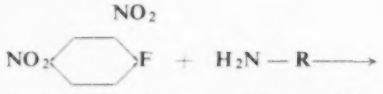


Fig. 10. Turntable of the Autoanalyser, loaded with plastic beakers of standards and samples.

Chromatography.—This technique has made it possible to study the intricacies of many biological phenomena, which were quite impossible to investigate before because of the lack of any adequate means to separate, characterize and measure the very small amounts of substances, e.g. amines, amino acids, sterols, etc., which result from many metabolic processes. Any derangements of these processes were still less capable of study. Chromatography, and particularly paper chromatography, can achieve a separation of almost infinitely small quantities of metabolites, which are easy to identify on the paper and often to measure quantitatively. Fig. 13 illustrates reversed phase chromatography of DNP-amines on benzoylated paper, using chloroform- methanol-water as solvent. The dinitrophenyl derivatives of amine are formed according to the following equation:



2·4-dinitrofluorobenzene primary amine

DNP-amine

The separated DNP-amines run up the paper at

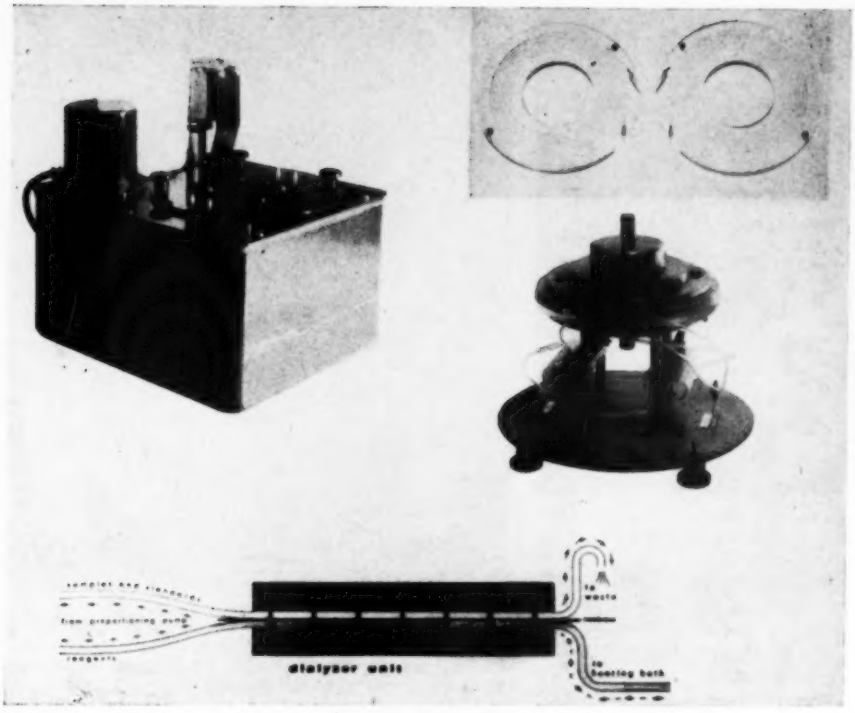


Fig. 11. Dialysing apparatus of the Autoanalyser.

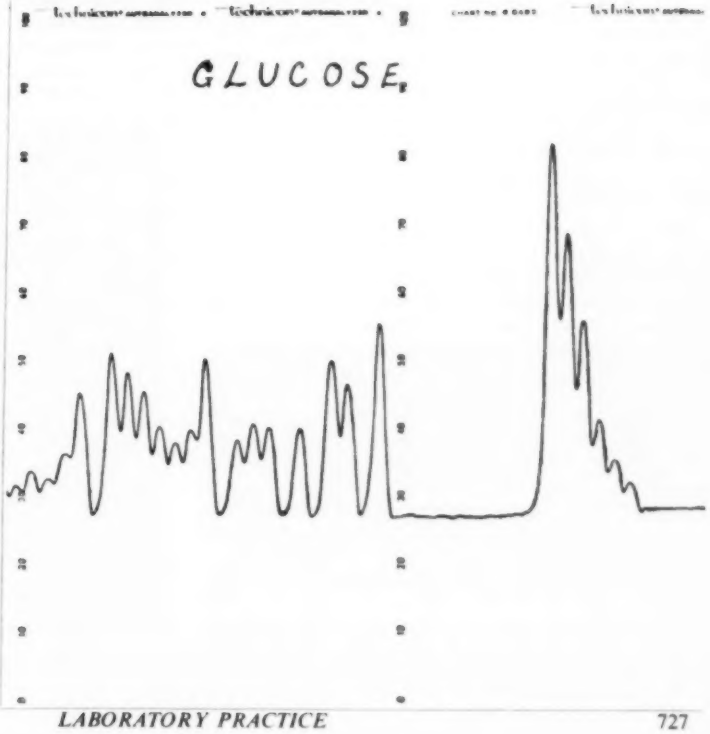


Fig. 12. Galvanometer recording of sugar standards and sugar concentrations of blood samples.

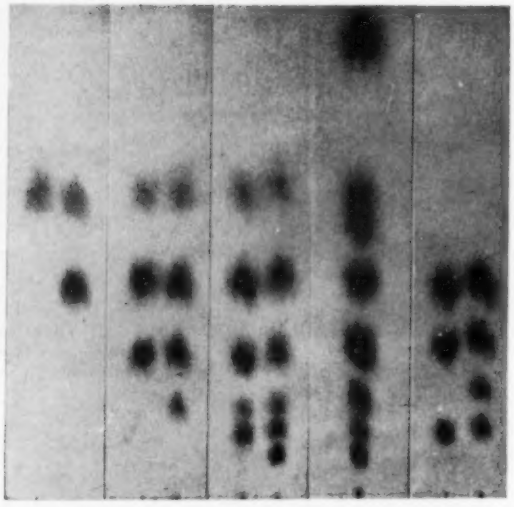


Fig. 13. Reversed phase paper chromatography of D.N.P.-amines. See text for distribution of spots.

different rates, those of low molecular weight, generally speaking, going fastest. The position of their spots on the paper can be revealed by photographing under ultraviolet light. In the figure the different amines appear in the following positions.

DNP-ammonia
DNP-methyl amine
DNP-dimethylamine
DNP-ethyl amine
DNP-n-propylamine DNP-diethylamine
DNP-butylamine Piperidine
DNP-isoamylamine
DNP-β-phenylethylamine

Increase in chain length slows down the movement of the compound.

Fig. 14 shows the DNP-derivatives of amines in urine. The reversed-phase chromatography of the urine and the reference substances was carried out on paraffin-treated paper with chloroform-methanol-water-paraffin as solvent. No DNP-ammonia appears because the ammonia had been removed from the urine by preliminary treatment with mercuric oxide. The first column of spots is that from a normal urine, the second from a pathological urine and third, fourth and fifth columns are of reference substances. By comparing it with Fig. 13, it can be seen that the position of the spots shows the presence of the DNP-derivatives of methyl-amine, di-methylamine, ethylamine, pyrrolidine and piperidine in both the normal urine and the pathological urine.

Fig. 15 shows a paper chromatogram of the indoles in urine from a patient with a rare tumour called argentaffinoma. It shows not only an abnormally large excretion of 5-hydroxyindoleacetic acid (5-HIA, upper centre large spot) but also spots of 5-hydroxytryptamine

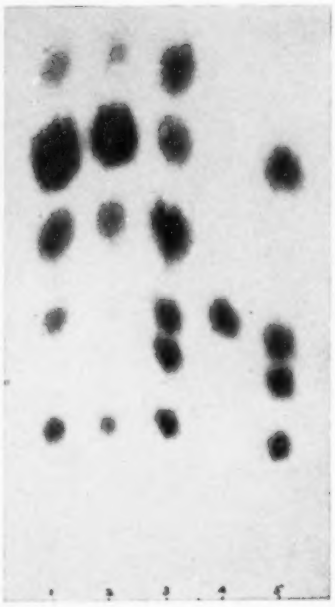


Fig. 14. Paper chromatogram of D.N.P.-amines in urine.

(5-HT, lower centre smaller spot) and 5-hydroxy-tryptophan (5-HTP, lowest spot). The 5-HT and 5-HTP are rarely encountered in urine from patients with argentaffinoma, a very unusual case. This chromatogram is an example of a so-called 'two dimensional run'; a butanol-acetic-acid-water solvent was allowed to run up the paper to carry the indoles in that direction. The

Fig. 15. Paper chromatogram of indoles in urine. Argentaffinoma. See text.

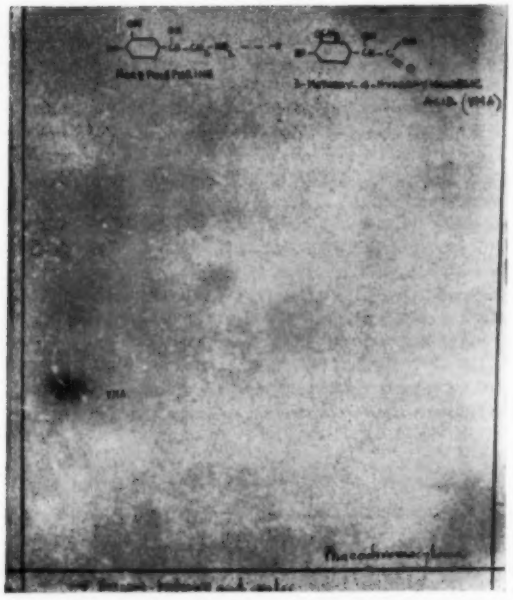


Fig. 16. Paper chromatogram of extract of urine from a patient with phaeochromocytoma, showing spot due to 3-methoxy-4-hydroxymandelic acid (vanillyl mandelic acid—V.M.A.), a breakdown product of epinephrine and norephinephrine. See text.

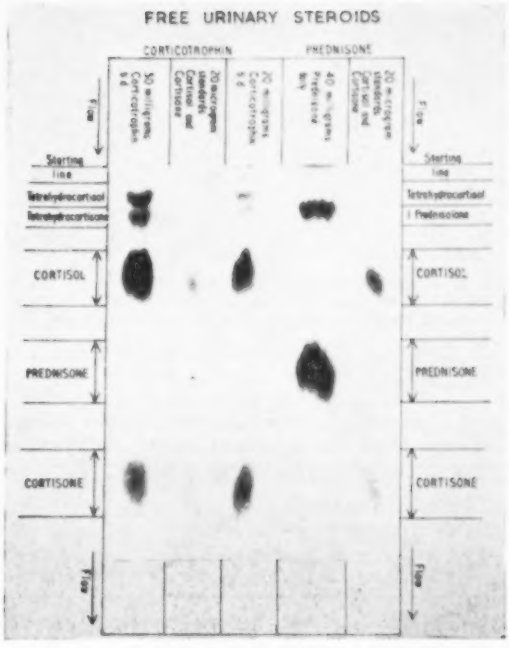


Fig. 17. Paper chromatogram of steroid hormones excreted in the urine. See text.

paper was then dried and a second solvent, 20 per cent KCl, allowed to run in a direction at right angles to that of the original solvent. This manipulation makes for a wider separation of the substances on the paper chromatogram. The presence of the indoles on the paper was revealed by spraying it with Ehrlich's aldehyde reagent. Fig. 16 is a paper chromatogram of the ethylacetate extract of urine from a patient with phaeochromocytoma. 3-Methoxy-4-hydroxymandelic acid (vanillyl mandelic acid-VMA) is a breakdown product of epinephrine (adrenaline) and norepinephrine (noradrenaline). VMA is increased in phaeochromocytoma. In the chromatogram the spot corresponds to about 9 μg. of VMA per mg. of creatinine; this is about four and a half times normal. Before the use of paper chromatography to identify this breakdown product of epinephrine and norephinephrine, it was customary, and in many laboratories still is, to estimate these two catecholamines themselves, a tedious and a not very accurate process. The solvents used were isopropanol-ammonia-water and benzene-propionic acid-water. The paper was sprayed with diazotized *p*-nitroaniline which reacts with the 3-methody-4-hydroxymandelic acid.

I am indebted to Dr. A. M. Asatoor for these paper chromatograms.

Steroid substances.—Fig. 17 is a paper chromatogram of an entirely different set of substances, i.e. the steroid hormones excreted in the urine. *Lane* 1 is an ACTH urine extract. 30 mg. of administered corticotrophin induced the excretion in the urine of tetrahydrocortisol, tetrahydrocortisone, cortisol and cortisone (ref. *Lane* 2), while 20 mg. of corticotrophin caused the excretion of lesser amounts (*Lane* 3). In *Lane* 4 it is seen that the administration of 40 mg. prednisone completely suppressed the excretion of the adrenal cortical steroids, cortisol and cortisone (ref. columns 2 and 5).

This figure was kindly furnished me by Dr. C. L. Cope.

Electrophoresis. Paper electrophoresis.—Paper electrophoresis of plasma proteins is well known. The technique

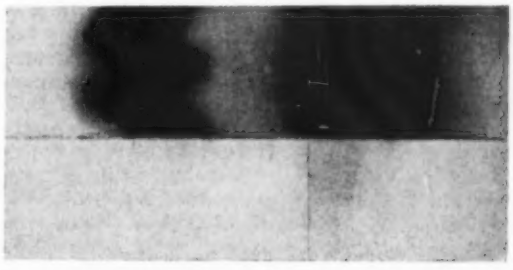


Fig. 18. Paper electrophoresis of purified alkaline phosphatase from placenta. Upper strip shows presence of two bands of protein. Lower strip shows presence of one band of phosphatase stained with *p*-nitrophenol from *p*-nitrophenylphosphate.

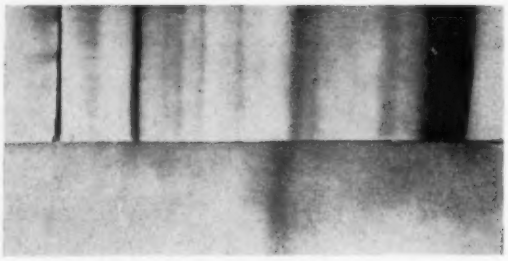


Fig. 19. Electrophoresis of blood plasma on starch gel. Upper strip shows multiple bands of protein. Lower strip shows single band of phosphatase in the β-globulin region.

is equally useful for separating and characterizing highly purified protein materials such as enzymes. Fig. 18 shows a paper chromatogram of highly-purified alkaline phosphatase from the human placenta (Ahmed and King, 1960). The upper band, stained with lissamine green, shows the presence of two bands of protein; the lower band, stained for the presence of phosphatase with *p*-nitrophenol phosphate (which is hydrolysed by the phosphatase to free *p*-nitrophenol) shows that only the right-hand protein band is enzymically active. Fig. 19 shows a separation of all the plasma proteins on a starch-gel electrophoretic strip. The separation on starch-gel is much sharper than it is on paper, and staining of the proteins with amidoschwartz reveals the presence of many more, sharply defined, bands than are seen on paper electrophoretic strips. The second strip in this figure is stained for phosphatase, and shows an extraordinarily sharp separation of the enzyme even though present in minute amounts, from an enormously greater amount of accompanying enzymically-inert protein. In this strip the phosphatase was revealed by its hydrolysis of α-naphthyl phosphate, the liberated free α-naphthol being coupled with tetrazotised ortho-dianisidine.

(To be continued)

BOOK REVIEWS

ICLA Symposium, 1958: Living Animal Material for Biological Research. Surrey: The Secretary-General, International Committee on Laboratory Animals, Woodmansterne Road, Carshalton. pp. 100 + iii (mimeographed). £1.

There is no date of publication on this, the proceedings of the Symposium sponsored by UNESCO and held at Gif-sur-Yvette on October 6-9, 1958, but is presumably May, 1960, or nearly two years after the event, a factor that detracts somewhat from its value and is difficult to understand in view of the relatively few papers (13, mostly short) and of the reproduction of small portions only of what are stated to have been full and fertile discussions.

There were 26 delegates from 15 different countries—this in itself must have complicated the tasks of compilation and editing—but the papers and discussions are mainly in English and partly in French. As might be anticipated, almost all the proceedings dealt in general terms with the problems discussed, but they are none the less important on this account. Much remains to be done in this country, and doubtless more in some others, to propagate the doctrines of good laboratory animals husbandry and usage.

On the first day of the Symposium, seven papers were presented dealing with present needs of cancer research, microbiology, tissue and cell cultures, endocrinology, physiology, radiobiology and the pharmaceutical industry. On the second day, reports were received from the heads of national laboratory animal organizations and from others competent to speak on surveys on the production and utilization of laboratory animals, and 16 countries were covered in this way. In view of the material already published by ICLA, however, this session is not reproduced in the present volume. On the third day, the work of ICLA (International Committee on Laboratory Animals) since its inauguration in December, 1956, was discussed, 'in an attempt to estimate the relevance of work already undertaken to the tasks confronting it.'

In view of the now widespread belief that our Russian colleagues enjoy excellent facilities, it is interesting to read in Prof. H. E. Pogosianz's paper, 'On the introduction of new species of animals into laboratory use', that 'We keep steppe-lemmings (*Lagurus lagurus* Pall.) in glass mouse jars with some sawdust and hay on the bottom', and that 'Some individuals have survived in our laboratory for about two years, but the number of those reaching natural death was small owing to lack of adequate space.'

The reviews of the work of ICLA and of national surveys so far completed, both by Dr. W. Lane-Petter, and the paper on 'Service function of mammalian genetics to biomedical research' by Dr. Elizabeth S. Russell, are of particular interest and importance.—

ALASTAIR N. WORDEN.

Mechanical Design of Laboratory Apparatus, by H. J. J. Braddick, B.A., Ph.D. London: Chapman and Hall Ltd. New York: Reinhold Publishing Corporation. 1960. pp. 47. 6s.

This booklet of some 50 pages deals primarily with the application of kinematic principles to design and is one more of the series of Monographs published by the Institute of Physics which are intended for students taking courses for the Higher National Certificate in Applied Physics. However, the preface indicates that the author has set out to reach a wider field of reader and in this he may not have been completely successful. The student of physics will have no difficulty in understanding the vocabulary used but the introduction of a

few explanatory paragraphs (or footnotes) here and there would considerably simplify the book for these additional readers.

The booklet is divided into three chapters of which the first and third are extremely short. Some very sound and useful advice is contained in the introduction which ends with a sentence which will have the fullest support from innumerable long suffering workshop technicians. All too frequently an inherently bad design has been saved by otherwise unnecessarily precise workmanship.

Of the 30 pages of the second chapter some 15 are given over to consideration of kinematic principles. This enables the subject to be dealt with quite extensively but some of the terms used in the earlier explanations may not be immediately intelligible to the student less advanced in physics. The next five pages deal with the application of kinematic principles and include a number of well-known examples. Then follow three pages on spring constraints with very brief sections on subassemblies, gears and shafts. The chapter ends with a section on connections between moving parts. No mention is made of the use of seals on rotating or sliding shafts or other connections. Perhaps a later edition might add some details of the more simple applications of 'O' rings to such purposes. Similarly, the omission of any note on belt or light chain devices, so useful in this type of laboratory apparatus, might be reconsidered.

The last chapter of the book is disappointingly short, being little more than two pages in length. Without attempting to compete with the many booklets on workshop practice which already exist, there would still appear to be room for considerable useful expansion. The use of metal castings is mentioned but not of cast plastics which are now extending their field of application. A note could usefully have been included on the effects of strain release occurring when machining both metals and non-metals. In this connection the production of stable flat baseplates on which to mount subassemblies has been known to produce difficulties for the inexperienced.

The absence of notes on the use of plastics generally, and on laminated plastics and Perspex in particular is to be regretted. Even where the final product must be made up in metal it is often worth while to construct a transparent prototype or mock up in order to be able to see what goes on inside.

The booklet is concisely written and well illustrated with diagrams. It goes some way to fulfilling a requirement, but the third chapter, merited a few more pages.—S. F. SUFFOLK.

Chemical Age Directory and Who's Who. 1960. 303 pp. 63s.

This enlarged edition contains lists of chemical organizations, D.S.I.R. and research association addresses, research institutes, administrative groups and, a new feature, particulars of the chemical and allied departments in British universities and technical colleges. The 'Who's Who' section now contains over 1,000 names. This is a most useful part as many of the persons concerned do not appear in the directories of the professional societies. Telephone numbers would increase the usefulness.

In view of the fact that business executives receive several diaries at Christmas, and this directory does not appear until May, we suggest that this item be deleted in future and further useful information be put in its place. For example a list of independent consultants classified under subjects would be very helpful to business men. The list of suppliers of chemicals could well be amplified and we trust that all the firms listed really do make or supply the chemicals mentioned. The list of suppliers of plant seems rather restricted. A master index of addresses and telephone numbers completes a most useful reference book.—J. G. DAVIS.

The Human Element in Research Management, by B. E. Noltingk, Ph.D., F.Inst.P., A.M.I.E.E. Amsterdam, London, New York, Princetown: Elsevier Publishing Company. 1959. 9s. 6d. pp. 91 + vi.

It is a matter of conjecture among the rank and file of some laboratories, whether the higher echelons of management have studied their methods in Machiavelli's 'The Prince', or whether they have evolved the same techniques for themselves. But however attractive some of the tactics appropriate to mediaeval Italy may appear to modern despots, there can be no doubt about their abhorrence by those on whom they are practised. Dr. Notlingk is firmly of the opinion that sweet reasonableness in research management is more appropriate than Machiavellian duplicity, to an extent unusual in this age, when reasonableness in management is often advocated as merely one facet of enlightened self-interest.

This little book crystallizes the musings of a scientist about the management of scientists, and a very good little book it is. In the first place it is, for a scientist, delightfully easy to read: it uses his own jargon instead of an alien one, and does not need the glossary which is seldom provided in books on management. In the second place it is concise, a great merit in a subject where verbosity appears to be rated a virtue. In the third place it contains, or so it seems to this reviewer, a great deal of close-packed common sense, and this despite a modest, almost apologetic, preface.

One telling device of the author's helps greatly to put across his message without waste of words. This is his free use of little pseudo-scientific graphical diagrams to show the connection between dependent variables. These are none the worse for being a form of jargon, for although their meanings are immediately apparent to a scientist, they might be stumbling-blocks to a non-technical reader. The idea of charting intensity of knowledge against a subject index such as the Universal Decimal Classification is a delightful example.

Dr. Noltingk writes of management practice, so he

says, without many years' opportunity to put all his precepts into practice. He believes that management of research is so much a question of handling personnel, that a deep understanding of the point of view of the man below is even more important than long experience of heavy responsibility; and that those who have not been 'men below' for many years may have forgotten some of the feelings that then excited them. That he has himself had the right blend of experience to warrant the writing of a book such as this seems to be verified by the result, which keeps in focus both the problems of the laboratory director, and the aspirations of those further down the ladder.

This is not a text-book on management; it raises more questions than it answers, and it is designed to set its readers thinking. Where solutions are suggested, they are, the author says, opinions rather than experimentally verifiable facts. As a stimulus and guide to thinking it is to be thoroughly recommended to all who practise management in research, from directors to junior scientists with one or two assistants. Buy it and read it, and then slip it on to your chief 's desk or into his brief-case.—ROSTRON HINDLEY.

An Introduction to Human Blood Groups, by Fulton Roberts, M.D. London: William Heinemann Medical Books Ltd. 1960. pp. 85 + viii. 9s. 6d.

The author is to be congratulated on producing such a concise and well-written account of the human blood groups. After a brief introduction to the principles of immunity, he discusses the immunological response to the Rhesus factor and its significance in haemolytic disease of the newborn—this leads to a discussion of the Rhesus complex, which is used as an illustration of the principles underlying the inheritance of blood group factors.

Thus introduced to the serology and genetics of blood groups, the reader is better able to understand the more complex notation, inheritance and reactions of the M.N.S., A.B.O., P. and Lewis blood group systems, which are discussed in that order.

Although lucidly described, their complexity makes one sympathize with the author's mild statement that 'the need for a revised notation for all blood groups is becoming pressing'.

Three short chapters at the end are devoted to the relationship between blood groups and disease, blood groups and genetics, and blood groups and anthropology.

This small book is highly recommended, for in its 85 pages it provides as clear an introduction to human blood groups as the current notation will allow, and discusses their clinical significance in the cause of disease, and the information they provide in understanding human genetics and anthropology. The book's clarity and brevity should give it wide appeal to both clinicians and students, for whom the aura of mystery which surrounds 'blood groups' should be removed.—M. C. BRAIN.

The Analysis of Sodium Potassium and Calcium in Biological Materials

(*Concluded from page 709*)

(*g*) The sodium and potassium concentrations in the 'calcium' solution were therefore 0·72 and 13·14 mg./100 ml. respectively. The interference correction to the calcium reading was calculated from equation V as —1·9 s.u. This was made up of —1·5 from part A of the correction and —0·4 from part B of the correction. The corrected calcium reading was 78·1 s.u., equivalent to 3·92 mg./100 ml.

(*h*) The corrected potassium and calcium concentrations in the 'sodium' solution were therefore 13·14 and 3·92 mg./100 ml. respectively. These were sufficiently close to the uncorrected values which were used for adjustment of the sodium reading, and no re-calculation was required.

The following comments may be made on these correction procedures. Firstly, the order of correction is important, because it is necessary to start the process by using the uncorrected concentrations of some of the interfering elements. In our example the largest corrections are to sodium, and so it was adjusted first. The other corrections were made using corrected concentrations of the interfering elements and did not change the potassium and calcium values sufficiently to affect the original correction to sodium. In a few instances, when both sodium and calcium have suffered large corrections, it has been found necessary to repeat the correction process, using the first set of corrected values in place of the uncorrected values. Secondly, the process is facilitated by using tables drawn up from the equations. As no very compact table can be made to represent equation V, part B, we have found it convenient to use a chart in this instance (Fig. 1).

ACKNOWLEDGMENT.—*We thank Mr. E. D. Goodall, who operated the flame photometer in all the experiments.*

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GLASS RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

To provide the glass industry with basic data on glass and glass-forming systems, the National Bureau of Standards carries on a programme of fundamental studies of the nature of glass. This programme is concerned with the structure and properties of glass, the behaviour of glass in unusual engineering applications, and the development of new glasses for special purposes. The primary objective of this work is to increase knowledge of the properties of glass so that it may be better utilized and find application in new products.

GLASSMAKING has been practised since ancient times. With the discovery that it could be blown into any desired shape on the end of a pipe, glass became of commercial interest. Although glassmaking by hand has now given way to automatic mass production techniques, the development of new glasses still remains almost as empirical as it was in ancient times. Only recently have scientifically rigorous studies been made of the structure and properties of glass.

Thus, the fundamental nature of glass structure is still not clearly understood. A number of structural models have been proposed, but none of these models has been completely verified by experimental evidence. Indications are only that there is a short-range orderliness of structure. Not only this problem of structure but numerous others concerning the properties of glass are under study by many scientists today. With improved understanding of structure should come increased ability to design glasses having specified optical, mechanical, thermal, and chemical properties.

To provide the basic data needed for these studies, the National Bureau of Standards has been conducting a programme of fundamental research on the properties and structure of glass and glass-forming systems. This programme, under the direction of C. H. Hahner, is concerned with the glass-forming process, the nature of the vitreous and supercooled states, viscosity and surface tension at elevated temperatures, the strength and elastic moduli over a wide range of temperatures, surface properties, and the bulk structure of glass. Other closely related investigations deal with the behaviour of glass in unusual engineering applications, the development of special glasses for precision optical instruments, and standard glasses with closely measured physical properties.

Interpretation of Glass Stability

Fundamental studies of glass-forming regions of simple ternary systems are providing an increased understanding of the nature of glass. For example, analysis of glass stability in terms of phase diagrams has shed additional light on the glass forming process and on the structure of multi-component glasses. This work contributes to present glass theory by indicating that distinct chemical entities exist in glass. Of practical use as well is the related finding that stable glasses are usually found in areas of a phase diagram where the liquidus curve or surface is flat—an indication of a highly dissociated compound.

As stable glass formation is directly opposed to crystallization or devitrification, studies of glass stability include a determination of the factors affecting crystallization. One is the liquidus temperature—the highest temperature at which molten glass and the crystalline phase can coexist in equilibrium; another is the rate of crystal growth at various temperatures; a third is the seeding or nucleation time required for crystal growth to start. A comparison of liquidus data with observed glass-forming ability provides a basis for structural interpretation.

Excess Entropy of Glass

Studies of the excess entropy of glass should also provide a better understanding of the vitreous state. Excess entropy is a measure of the greater molecular disorder of glass as compared with corresponding crystals of the same composition. Efforts are under way to measure this excess entropy in two ways on glasses for which stoichiometrically corres-

Oscillating cup viscometer developed at the National Bureau of Standards for measuring viscosity and surface tension of glass at temperatures above 2,000° C. In the upper foreground is the vacuum chamber, raised to show the induction heating coil surrounding the oscillating cup. Adjustments on the vacuum system are being made in the lower foreground. In the background is the high frequency generator.

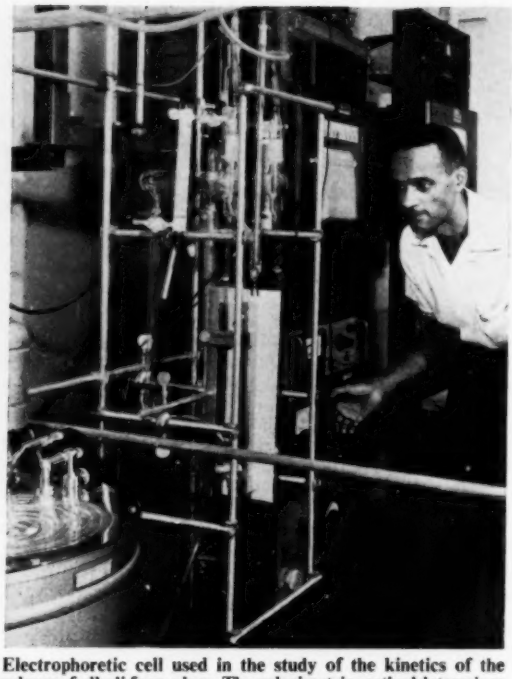
A cathetometer is being used to read the mercury level in a dilatometer immersed in a thermostating bath. The device is used to measure thermal expansion of glass at and near room temperature.

ponding crystals also exist. One method is by direct measurements of specific heats over the temperature range from just above the melting point down as near as possible to absolute zero. The entropy loss thereby determined over this range for the glass, as compared with that for the crystal, gives values for the residual entropy. A precision low-temperature adiabatic calorimeter for part of these measurements is nearing completion. It will be supplemented with a high-temperature drop calorimeter. In the second method, vapour pressures are being measured as a function of temperature for substances in vitreous and crystalline states. The entropies of vaporization obtained from these measurements give values for the excess entropy of a particular specimen of glass. Such measurements on vitreous arsenic trioxide have led to interesting side-results indicating an extremely low condensation coefficient.

Glass Transition Temperature

As a criterion for the transformation between the vitreous and the supercooled liquid states, glass transition temperatures are being investigated. The effect of rate of temperature change on the volume-temperature curve is being measured for experimentally convenient glasses such as nitrate mixtures, selenium, and glucose, which have glass transformation temperatures in the vicinity of room temperature. Variations in rate of temperature change result in variations of transformation temperature of less than 6 °C. for a given composition. Measurements of glass transformation temperature as a function of composition in two-component systems have suggested that certain systems form glasses only at stoichiometric ratios of the compounds, and a greater number from glasses at nonstoichiometric ratios.

Measured differences in thermal expansion coefficients for glass-forming substances above and below their glass transformation temperatures have been compared with values predicted theoretically from their viscosity-temperature relations. Results indicate that in most glasses, including silicates, the molecular structure may not change drastically with temperature in the transition region. However, in borate and nitrate glasses the thermal expansion data do not conflict with the concept of structural changes.



Electrophoretic cell used in the study of the kinetics of the release of alkali from glass. The calorimetric method determines the amount of alkali released.

Transport Phenomena

The study of transport phenomena in vitreous silicates gives important information on both the bulk structure of glass and the structure of the surface domains. As many properties of glass are highly dependent upon the surface condition, this area of investigation promises to yield much information of both theoretical and practical value. Under study at the present time is the migration of mobile ions, such as alkali, through the glass under the influence of a concentration gradient created by subjecting a glass surface to water or other environments containing diffusion sinks for alkali. The experimental study of the diffusion kinetics is performed by a coulometric method that simultaneously determines the amount of alkali released and removes any corrosion products, to maintain a constant pH. The data obtained are compared with mobility data derived from electrical conductivity measurements and analyzed with respect to various theoretical diffusion models. Of further interest are the details of the exchange-mechanism in this process; in particular, how the water constituents enter the structure of the glass. Methods are being developed not only to observe the kinetics of the net-diffusion process but also to obtain successive concentration profiles through the glass at various stages of the diffusion process.

High-Temperature Viscosity and Surface Tension

One phase of the study on the properties of glass is the development of methods for measuring viscosity and surface tension at temperatures up to 2,000 °C. Such data on liquids at high temperatures are useful both for advance of production methods and for progress in fundamental scientific

Rotating cylinder viscometer for measuring the viscosity of glass at temperatures up to 1,450°C.

This high temperature drop calorimeter is used in studies of the excess entropy of glass.

Portion of the Bureau's glass research laboratory where the molecular structure of the glassy state is under study.

understanding. So far, a room-temperature model of an oscillating cup viscometer has been designed and built. It has been calibrated at this temperature with oils of suitable viscosity. A high-temperature version is now under construction and will soon be completed. The apparent advantage of the oscillating cup viscometer for very high temperature work is that liquids under investigation can be in a totally enclosed container while the rest of the system is in a vacuum. Also, the cup containing the molten material is of a simple geometric shape so that it can be easily fabricated if frequent replacement is necessary. The first material scheduled for study is B_2O_3.

Strength of Glass at Elevated Temperatures

Knowledge of the mechanical properties of glasses at elevated temperatures is necessary for aeronautical applications. The Bureau is measuring the modulus of rupture and the modulus of elasticity from room temperature to a temperature near the softening point of the glass. The study includes the effect of prolonged heating on the strength and on the rate at which samples of both annealed and tempered glass deform under load. Most of the work is being carried on at the Bureau with the close co-operation of the Materials Laboratory of the Wright Air Development Centre and three glass companies: Libbey-Owens-Ford Glass Co., Corning Glass Works, and the Pittsburgh Plate Glass Co.

Only those glasses that are available in commercial quantities, or that have been produced in at least pilot-plant quantities in the form of polished plate, have been chosen for the study. Although the glasses selected for study are few in number they do cover a wide range of physical properties, particularly in respect to coefficient of expansion and strain point.

A study of test methods and methods of sample preparation has been completed. Strength tests on six types of glass—soda-lime-silica, 2 borosilicates, alumino silicate, 96 per cent silica, and fused silica—are almost complete.

Special Optical Glasses

The increasing use of infra-red devices in the fields of target detection, fire control, missile guidance, and aerial reconnaissance creates a demand for improved optical materials

for their construction. To develop multi-component glasses having particular infra-red transmission properties, the Bureau has been investigating the size, shape, and location of the glass-forming areas in the phase diagrams of simple two- and three-component systems. The physical and optical properties of the simple glasses can be correlated with composition to furnish clues to the atomic and molecular units composing the glass structure and their arrangement.

The effects of small amounts of water in the internal structure of glass have been largely neglected in the past. The presence of this water is strongly evident in the infra-red spectra of practically all glasses. If glass is to serve as an infra-red material at wavelengths beyond 2·75 μ, means must be found to eliminate, or reduce, the water content. Work in this area has been in progress for some time. Glasses have been made in silicate, germanate, calcium aluminate, and tellurite systems, and many of their properties determined.

Multi-component glasses based on the BaO-TiO_2-SiO_2 system have been made having good infra-red transmittance, refractive indices up to 1·94, deformation points above 800°C. and excellent chemical durability. Similar germanate glasses have even higher refractive indices, transmit to somewhat longer wavelengths, deform above 700°C., and also have excellent chemical durability. Calcium aluminate glasses transmit to about 6 μ except for a deep absorption band near 2·8 μ. They have refractive index values of about 1·67 to 1·70, and deformation points near 750°C. However, they tend to devitrify easily and are attacked by moisture. The glasses based on TeO_2 have high values of refractive index, some of them being above 2·00. They deform at 350 to 400°C. and have high coefficients of thermal expansion, which makes them sensitive to thermal shock. The glasses devitrify easily and evidently cannot be cooled in large amounts free of crystalline material.

Recently it has been found that the water band absorption is greatly reduced by the use of fluorides as an ingredient of the batches from which the glasses are melted. While the method has been used only on a laboratory scale, it appears to be usable on melts of any size.

Standard Sample Programme

The Bureau has for a number of years made available standard samples of glass to be used as checks on the accuracy of chemical analysis of glass. Two additional types of standard samples are now being considered. One will be a series of glasses, representative of important commercial types, that can be used to calibrate and standardize instruments for measuring the properties of glass. The second will be a series of high purity containing one, two, or three components. These glasses will be used to compare results between laboratories especially in studies of structure and constitution.

A soda-lime-silica glass which is representative of a widely used commercial composition is now being studied for use as a standard of viscosity and surface tension for molten glass. In the lower temperature range up to 1,450°C., a calibrated rotating-cylinder viscometer is used. The surface tension of this glass will also be determined over a similar temperature range. Additional types of glass, representative of those commercially available, will soon be obtained to extend the work on viscosity and surface tension calibrations as soon as the determinations on the soda-lime-silica glass are complete.

REPRESENTATIVE PUBLICATIONS IN GLASS RESEARCH BY NBS AUTHORS

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Society for Applied Bacteriology

The Annual Meeting of the Society for Applied Bacteriology was held in Bangor on July 5. The following are now officers of the Society.

Hon. President: Mr. G. Sykes.
Hon. Secretary: Dr. Ella M. Barnes.
Hon. Treasurer: Mr. G. Elis Jones.
Hon. Editors: Dr. S. E. Jacobs and Dr. D. J. Jayne-Williams.
Hon. Publications Manager: Mr. A. H. Walters.
Hon. Advertising Manager: Mr. E. J. Mann.
Members of Committee: Dr. Eve Billing, Mr. N. J. Butler, Mr. B. M. Gibbs, Dr. N. D. Harris, Dr. J. R. Norris, Dr. Jane Meiklejohn, Dr. A. M. Paton, Dr. Joan Taylor, Dr. G. C. Ware (ex officio) Dr. C. A. E. Briggs, and Mr. D. A. McKenzie.

Laboratory Notebook

THE wide range of research topics in modern physics covered by the work of the National Physical Laboratory is described in the latest Annual Report of the Laboratory (H.M.S.O., price 8s.)

In the new Basic Physics Division the programme has been aimed at investigating different aspects of the physics of polymers, by means of the most modern techniques for examining the atomic structure of matter. Success in understanding how the atoms are held together in plastics and other polymers, and how these arrangements of the atoms define their mechanical, electrical and thermal properties would have far-reaching consequences. Only a beginning has been made so far.

New work is also reported from the CME Division, recently renamed the Autonomics Division. A start has been made on the problems of mechanical translation from Russian into English, using the ACE computer. Work of special interest to industry is concerned with methods for self-optimization of industrial control processes; as a first example of this the operation of a distillation column is being simulated on a computer.

The programme of the Aerodynamics Division covers many aspects of research of importance to future aircraft and missile design and development. The phenomena of buffeting and aileron buzz, which decrease the safety and controllability of aircraft when they occur, have been studied. As a result means of suppressing these effects have been suggested. On the non-aeronautical side studies of the airflow around tall chimney stacks have suggested methods of eliminating 'flutter'. One successful idea is to wind helical 'strakes' round the chimney.

The Standards Division has continued to foster international collaboration in several fields. In June last year the new International yard and pound already legalized by Canada and the U.S. were adopted for scientific purposes in this country. Determination of the density of mercury has been completed by measurements on samples from the standards laboratories of Australia and the U.S. A start has been made on correlating U.K. and U.S. time and frequency services. The scale of temperature between 10°K. and 90°K. defined by the platinum resistance thermometer has been related to the thermodynamic scale by means of a helium gas thermometer. Comparisons are being made with the U.S. and Russia, with the intention of extending the International Temperature Scale below its present lower limit.

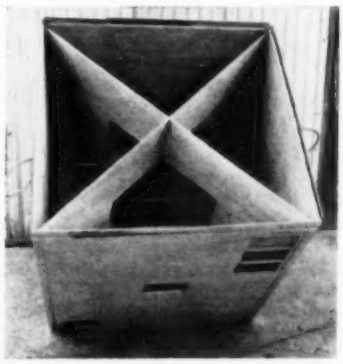
Spectro-radiometric methods have been tried successfully by the Light Division for the first time in the establishment of the standard scale of colour temperature. Emphasis has also been given by the Division to improving diffraction gratings for metrological purposes in industry, particularly in association with manufacturers from the machine tool industry.

The Metallurgy Division which is now equipped with some of the best modern research tools available for the study of metals (including an electron microscope, soft x-ray spectrograph, mass spectrograph and optical spectrograph), is bringing these modern techniques to bear on some of the problems associated with precipitation processes in iron. It is now possible, by using the electron microscope, to see dislocations (i.e. the faults in the atomic planes which weaken metals by one hundred to a thousand times).

The new facilities for ship hydrodynamics research which were opened at Feltham by H.R.H. The Duke of Edinburgh a year ago will allow studies to be made of the seakeeping properties of ships, of the influence of hull shape, anti-roll and anti-pitch fins, etc., on the motion of ships in calm and rough waters. In conjunction with the Mathematics Division, the application of high-speed computers to ship design has been started.

Containers for Radioactive Materials

Increased use of radioactive materials in hospitals, factories and laboratories has brought a whole new range of packaging problems for those concerned in shipping and delivering these materials throughout the world. One particular difficulty is the degree of packaging protection needed to ensure that radioactive components are safe to handle—invariably the size and weight of the pack have to be out of all proportion to the size of the elements themselves. Use of the lightest and least bulky packaging materials available is therefore even more than usually important.



Venesta plywood cases used as outers for the transport of radioactive materials.

The lightness and rigidity of thin-wall plywood packing cases has been turned to special advantage in an unusual packaging operation recently completed by Lep Transport Ltd.

The product to be packed was a tiny 'thimble', not much bigger than the household article but radioactive to a degree that prevented any more than eight being bulk-packed together. Moreover, to give a safety factor, the bulk pack of eight had to travel in a container large enough to leave a thick, insulation cushion of air on all sides.

For this reason, the packs were made up of no less than four different-sized containers, one inside the other. First, the thimbles were individually packed to Ministry specifications in cartons measuring 1½ in. by 1 in. by 1 in. which in turn were put into specially-fitted cartons each 7 in. by 7 in. by 7 in. Eight of the larger cartons were then packed together in a solid fibreboard case 15 in. by 15 in. by 15 in.

Outer containers, selected by Lep themselves, were metal edged plywood packing cases of the standard Pattern 201N made by Venesta Plywood Ltd. Inside, the fibre case containing the cartons rested clear of the sides in a support formed by two cut-out cross panels. No other internal fittings or cushioning were needed.

To give the air space required, the plywood containers had to be more than twice the size of the fibre cases—in fact, 36 in. by 36 in. by 36 in.

pH Measurement of Papers

Research has been carried out at the Printing, Packaging & Allied Trades Research Association which will help to simplify the measurement of the pH of papers. One of the easiest ways to measure pH is to use the Lovibond comparator, in which an indicator is added to a water extract from the paper, the pH value being determined by comparing the colour produced with that of different coloured glasses in the instrument.

Many printers and papermakers bypass the extract stage and make a smear on the paper and then compare the colour of the stain with the comparator standard glasses. Although this can, in some instances, give results of value, there is a limitation in the pH range that can be measured; generally, pH values cannot be accurately determined inside the range 3·6 to 5·2—the critical values in ink drying problems. This is because the colour of the stain often does not match up with any of the colours on the comparator, due to the dichroic nature of the indicator solutions.

The colour standards were made with the sole object of providing a comparison for extract *solutions*, not for the

so-called 'smear' technique, and therefore errors are unavoidable.

The purpose of the PATRA experiments was to line up the comparator colours with those obtained by staining. With the co-operation of The Tintometer Ltd., Salisbury, a special set of standard glasses designed especially for those printers and papermakers using the smear technique has been developed. It is expected that these will shortly be available commercially.

Full details of the PATRA experiments are given in a report by D. H. Charlesworth and R. R. Coupe; it is available to members on request.

The authors conclude from the experiments that the difficulty of matching the colour stains can be overcome by having a special set of standards specially prepared. Because of the confusion that would be caused by using the same range of pH values for both the preferred standard extract method and the smear technique, they recommend a special index. These would range from 10 to 200; each step of 10 in the index would represent a pH difference of about 0·2 units, the figures below 100 becoming progressively more acid and above 100 more alkaline. This scale will be known as the Lovibond-PATRA pH Index.

The authors say that probably the greatest use of the new pH scale will be in connection with ink drying problems. As an approximation, uncoated papers should have a Lovibond-PATRA pH index of 100 or above. Similar specifications can be built up for other paper pH requirements.

The smear indicator method may be used by either papermaker or printer. For the papermaker who is using the test as a quality control on a standard line, it will prove satisfactory, for he can establish his own correlation between smear pH index and the primary hot extract standard.

Printers would be able to check successive consignments of the same paper or papers of similar manufacturing history, and would be able to tell whether papers would be satisfactory from an ink drying point of view.

Analogue-Digital Computing Centre

A new type of computing service centre, based on a machine using both analogue and digital techniques, is now in operation at the Computer Department of Redifon Ltd., Crawley, Sussex.

The machine for hire at the centre is the RADIC (Redifon Analogue-Digital Computing System), claimed to be the first of its kind in the world to combine the advantages of both analogue and digital computing techniques in a single system.

Introduced in June, 1960, the machine enables certain classes of calculation to be performed quicker and more cheaply than is possible with conventional computing methods. Cost of hiring the system is from £12 to £50 an hour, depending on the size of the computation. Personnel are available to assist in preparing and translating the customer's work for the machine.

Present work being undertaken at the centre includes: optimum blending of chemical products; calculating by linear and non-linear programming the best deployment of land and sea transport to minimize costs; calculations on heat transfer and related problems in chemical distillation columns; calculations on the design of pressure containers, and simulation and analysis of certain processes in paper making plant.

In the near future it is intended to transfer the computing service centre to the inner London area and at the same time extend the capacity of the present medium sized equipment.

Suggestion Box

Under a suggestions scheme run by The Morgan Crucible Co. Ltd., Mr. James H. Fairweather, 57, laboratory assistant in the company's Research Department, has been awarded £225.

His alteration to the cooling system of a laboratory furnace will save approximately 10 million gallons of water a year, and represents an annual saving to the company of £400.

Mr. Fairweather's success comes after 21 years with the company, which, already this year, has paid out £790 to employees under the scheme.

News from the British Association of Chemists

A U.S.S.R. Research Institute

By H. WARSON, B.SC., F.R.I.C.
(Hon. Editor of the B.A.C. Journal)

DURING a visit to the International Symposium on Micromolecular Chemistry, which took place in Moscow from June 14-18, I was fortunate, largely through the assistance and patience of one of the symposium interpreters, in being able to visit the Institute of Organic Chemistry.

This is a handsome multi-storey building in the Prospekt Leningrad, in the south of the city, in an area which is largely devoted to research buildings, and other cognate activities allied to the University, which is in the same area. My guide and interpreter was Dr. N. A. Platé, and he was assisted by Prof. Kaverzneva and Prof. N. I. Shuikin.

The Institute was originally founded in Leningrad by N. Zerlinsky in 1935, but was later moved to Moscow where it occupied at first some laboratories in the University. One of the principal lines of research has always been the catalytic formation of hydrocarbons Prof. Favorsky was the first Director. The Institute was later divided into two separate Institutes, one dealing with physical-organic chemistry, and the other with organic chemistry alone. Academician Kazansky became Director of the Institute of Organic Chemistry in 1955. Other famous names in connection with the development of the Institute are Nesoreyanov and Demianov.

At the present time the Institute has 300 full-time research workers, and about 150-170 technicians and mechanics on its staff.

A visit to some of the laboratories proved of considerable interest. They are constructed on a standard unit basis on either side of a central corridor, access to services being through wooden panels in the walls of the corridor. Each unit has a width of about 10 ft. permitting the use of two facing benches along the length of the laboratory which seemed about 27 ft. deep. Windows are doubled because of the winter climate. Several laboratories were arranged in multiple units with island benches. They all seemed densely packed with miscellaneous apparatus, rather more so than in this country.

Laboratory Apparatus

Most of the more specialized physical apparatus is of Soviet construction, and I was shown a gas-liquid chromatography apparatus, with a thermal conductivity detector. The laboratories of Prof. Kaverzneva, who is working on protein chemistry contain an automatic fractionation apparatus. Prof. Shuikin, who stressed that the place for a chemist was in the laboratory and not at a desk, has extensive laboratories for the study of hydrogenation and hydro-

genolysis. He has given a number of papers, some published in English, at International Symposia. Semi-scale pressure plant for hydrogenation is contained in the ordinary laboratories in a safety cage, and is not considered to present undue risks.

Organic Chemistry Laboratories

Other laboratories in the Institute cover a wide field of organic chemistry. The Institute can be considered in some ways analogous to one of our own D.S.I.R. laboratories. While specializing in theoretical studies, these are expected to have some ultimate practical applications.

The Institute has an extensive library, with adequate space for study. The Western (mainly English) section is very complete, and would compare favourably with any comparable research station here. The Russian section contains the new series of Russian chemical abstracts which have been issued for about five years, and whose cover is gradually being extended to different branches of the science.

Salaries and Conditions

Before making any general comparison of salaries in Russia with those prevailing in Great Britain, it must be remembered that relative costs of essentials in the two countries vary widely, and are discussed to some extent below. For comparative purposes, the tourist rate for the rouble (28 roubles = £1 sterling) would give a reasonable comparison.

At the Organic Research Institute, the working day lasts from 10 a.m. till 5 p.m. with one hour for lunch. Some Institutes work a slightly longer day, but organic chemical work, like radiochemistry is considered to be in some degree deleterious to health. Staff at the Institute are graded, the raw graduate without research experience being known as an 'Aspirant.' After two years he submits a thesis, and thence becomes a 'Kandidat' or junior research worker, for a further period of about two years until he submits his thesis as a 'Kandidat,' which can be considered of Ph.D. standard. The final qualifying stage is that of a senior research worker, at the end of which period a degree equivalent to that of a D.Sc. is awarded.

While I have used the term 'he' for a research worker, it is worthy of note that there are far more women employed than is the case in the West. This does not apply to the junior positions alone, and it is evident that women chemists continue at their posts after marriage, as is indeed the case with most professions in the U.S.S.R.

An 'Aspirant' commences with a salary of 980 roubles per month (approximately £35). This rises in three instalments which depend on ability rather than the calendar from 1,050 roubles per month to 1,200 roubles and thence to 1,350 roubles, which is the salary a junior research worker should attain when he takes the 'Kandidat' degree. Having attained this his salary is 1,750 roubles, rising to 2,000 roubles. A senior research worker's salary will rise from 2,250 or 2,400 roubles with three approximately annual instalments to 3,000 roubles. A group leader would earn 5,000 roubles per month.

The technicians have a salary scale which rises from 740 roubles per month to 980 roubles. This grade would include glassblowers and skilled operators of apparatus rather than the 'technologists' in the Western sense who often earn more than chemists or 'pure' scientists in Britain.

Holidays are very generous, varying from 24 days per annum for technicians and junior chemists to 36 days for 'Kandidats' and 48 days for doctors.

Some Notes on Costs in Moscow

Accommodation standards are still well below those of Great Britain. Private houses are virtually unknown, and rent in the vast new apartments is charged by floor space, excluding kitchen and bathroom. Rent depends on salary, but is very cheap, and a senior scientist who rented 75 square metres (the floor area of a medium size three-bedroom house in Britain) would pay the equivalent of about £5 per month. This amount of floor-space is exceptional, the normal floor area and rent being less than half of the above.

Staple foods seem to be comparable in price with the West. Thus rye bread is about the same price per kg. as a 2 lb. loaf, the price for white bread being double. Cigarettes vary from 1r 40 to 4r 50 per packet of 20, but I am told that a quality resembling British cigarettes costs about 'Channel Island' prices.

Clothing is in general approximately double British prices, and quality seems poorer, although doubtless the rough quality is preferred for climatic considerations in winter. Man-made fibres are generally considerably above this level, but some items from natural fibres are rather cheaper.

Transport is not an expensive item. Bus fares are 45 kopeks (about 4d.) for any distance, and metro fares are 50 kopeks. Theatres including the famous Bolshoi, and cinemas have prices comparable with or below our own. Long playing records are the cheapest items in the Soviet Union, a 12 in. disc costing 10 roubles (7s.). Musical instruments are also very cheap. Tools are moderately priced and toys are good value by our standards.

Luxury foods, however, are extremely expensive. Chocolate is about 12 times British prices (although the excellent ice cream is about 1 rouble per block!). Fruit, such as oranges, cost about four or five times the British price.

Conclusions

It must be realized that the salaries and conditions described here are essentially those in an academic institute, and not in industry, although possibly the two are comparable. In the U.S.S.R. there is no doubt that scientists are an elite, whose conditions are among the most favourable in that vast country, and the normal standards are rather lower.

Nevertheless, so little is known about the U.S.S.R. here, that a visit such as mine, albeit a short one, was well worth while. One retains pleasant memories of the personal friendliness encountered, and of the courtesy of one of my interpreters, who made an extensive diversion (caused by the State visit of the President of India), to take me back to my hotel, incidentally in his own car, a Volga.

General News

Life Members

The following have been elected Life Members of the Association:

S. R. Hole. Elected Member 1923. (Birmingham Section).
R. A. Howard. Elected Member 1934. (London Section).
J. P. Lorains. Elected Member 1919. (Scottish Section).
W. Salmon. Elected Member 1923. (Birmingham Section).

Obituary

The decease of the following members is recorded with regret:

F. Arend. (London Section). Elected Member 1948.
D. G. Barkla. (Liverpool Section). Elected Member 1919.
T. W. Cheke. (Notts. & Derby Section). Elected Member 1919.
R. E. Forster. (Birmingham Section). Elected Member 1919.
S. L. Levy. (Liverpool Section). Elected Member 1942.
H. Tilsley. (Yorkshire Section). Elected Member 1942.
J. R. Wright. (Yorkshire Section). Elected Member 1950.

All correspondence and enquiries concerning this Association should be addressed to G. R. Langdale, Esq., The British Association of Chemists, Nomination Dept., 14 Harley Street, London, W.1. Tel.: Museum 7021.

LABORATORY EQUIPMENT & APPLIANCES

Direct-writing Oscillograph Recorder

A new direct-writing oscillograph is capable of recording at frequencies from d.c. to 1,000 c.p.s. and above, and provides a choice of eight paper speeds, from 5 in. a minute to 150 in. a second. It gives a well-defined trace at writing speeds in excess of 12,000 in. per second.

Designed as a 12-channel unit, the recorder is available with matched amplifiers and power supply units if required, and is able to record any varying function which can be translated into an electrical signal within the frequency range. It is therefore of value in a large number of industrial applications ranging from stress analysis by electrical strain gauge techniques to direct recording of electrical power supply characteristics.

The oscillograph will be particularly useful as a test instrument for electrical undertakings and manufacturers of heavy electrical equipment, and in engineering laboratories for measurement of high-frequency dynamic strain and for process analysis. It will also provide a useful multi-purpose recorder for university laboratories and technical colleges, in many instances allowing a transient tube image to be replaced by a permanent record.

Its applications also extend to military uses, for missile testing, gun performance tests and general telemetry equipment; and to medical uses in cardio-vascular and respiratory investigations.

The magnet block carries 15 pencil-type mirror galvanometers, specially made for the recorder by the manufacturers, the additional three channels serving for time or event marking, or as extra recording channels if desired. By the operation of a switch, two of these three galvanometers can be connected with the mains supply to give a reference frequency.

The oscillograph employs a compact-source ultraviolet lamp, recording up to 15 traces on six-inch-wide sensitized paper. The images are developed spontaneously within a few seconds.

Rolls of recording paper containing up to 200 ft. can be loaded through a front panel, and feeding is automatic once the edge of the paper has been placed in contact with the drive roller. A footage selector switch is provided which stops the drive after 2, 5, or 10 ft. of paper have been exposed, or to allow continuous running.

Paper speeds are selected by push-buttons on the front panel which operate solenoid clutches. The running speed is indicated by a light opposite the appropriate button. All the gears are permanently meshed, so that speeds may be changed during recording and there is a minimum of delay in reaching even the highest speeds.

The position of each trace on the paper can be simply adjusted through an opening in the top cover, using a screwdriver, while the resultant change on the paper is observed through the front window. If less than the full number of channels are in use, unwanted traces can be directed off the edge of the paper. To identify the traces, a sequential interruption of each trace provides identification of each channel at two-foot intervals, irrespective of paper speed.

Five types of pencil galvanometer are available with the oscillograph, the standard general-purpose type giving response up to 1,000 cycles with a tolerance of ±1 per cent and a sensitivity of 7·5 mA/cm. Other galvanometers permit recording of frequencies up to nearly 2,000 c.p.s.

The amplifiers and power supply units have been designed specifically for the oscillograph, and if required can be assembled into modules for desk or rack mounting.

(Manufacturers: Savage & Parsons Ltd. Watford, Herts.)

Temperature and Operation Recorders

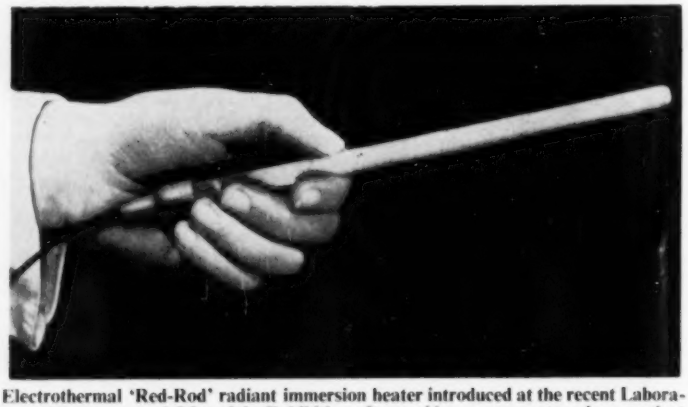
A new range of completely self-contained, portable temperature and operation recorders, designed to cover a wide field of applications in industry and in the laboratory, is now available.

Models are available for recording ambient temperature, for remote temperature recording, for operation recording (e.g. the on/off cycling of electric heaters, fans, etc.) and for the simultaneous recording of both temperature and operation.

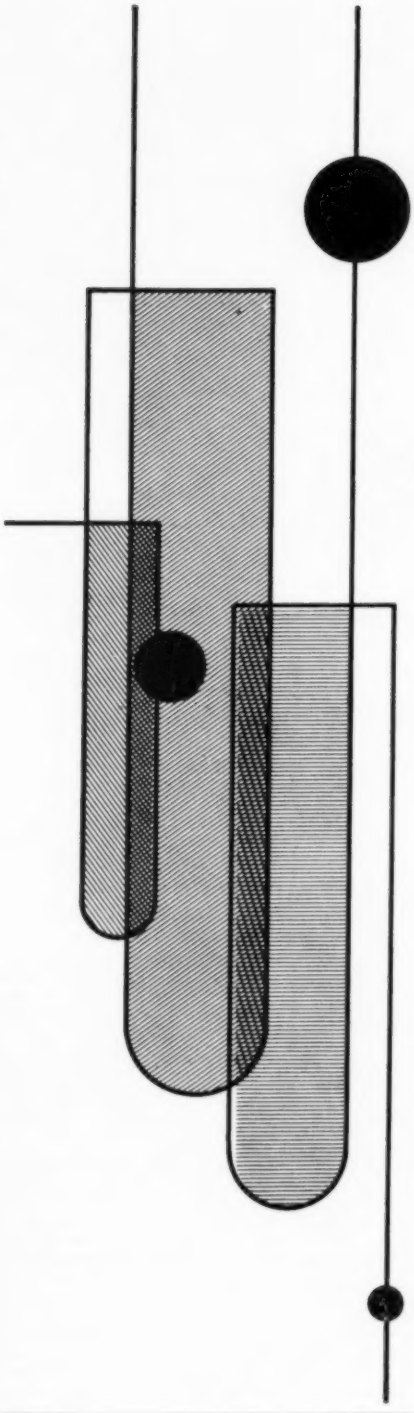
These new recorders follow the modern trend towards miniaturization of instruments, and measure only 5¼ in. wide × 4½ in. deep × 7½ in. high. They consist basically of two parts—the bakelite case containing the spring-driven clock mechanism, and a front panel or 'door' carrying the pen and actuating mechanism.

For recording ambient temperature, the door contains a bimetallic element. For remote temperature recording, a bulb-type element is connected to the door by 6 ft. of flexible capillary tubing (a special model with 10 ft. of tubing is

The 12-channel direct-writing oscillograph recorder gives impulses of frequencies up to 1,000 c.p.s.



Electrothermal 'Red-Rod' radiant immersion heater introduced at the recent Laboratory Apparatus and Materials Exhibition. It provides a compact and convenient source for the heating of liquids.



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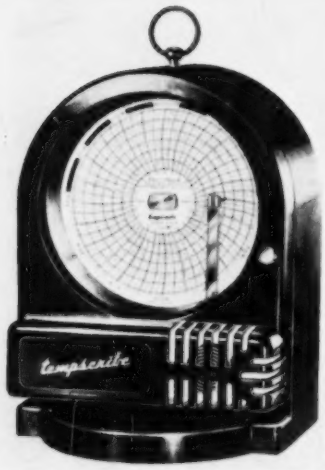

The new temperature and operation recorder.

available for biological refrigeration work, such as recording storage conditions of vaccine, serum, etc.); while for operation recording the door carries an electro-magnetic armature for parallel connection to the circuit concerned. The door for the temperature/operation recorder contains both a bulb-type element and an electro-magnetic armature, each actuating a separate pen.

An important feature of these recorders is that the doors are interchangeable and, within certain limits (imposed by the rotation time of the clock mechanism), any recorder can be converted to perform a different function, e.g. a temperature recorder may be converted to record over a different temperature range or to record electrical operations.

All models record on a circular chart 4½ in. in diameter. For recording ambient temperatures, charts are available covering 13 different ranges from —30 F. to +120 F. up to +100 F. to +160 F., over periods of four hours, eight hours, 24 hours or seven days. For remote recording there are six charts, with ranges from —30 F. to +120 F. up to +70 F. to +160 F. over periods of 24 hours or seven days.

For operation recording, three charts covering periods of four hours, eight hours or 24 hours respectively are available; and for combined temperature/operation recording two charts, both for a 24 hour recording period, with temperature ranges of —30 F. to +50 F. and —30 F. to +120 F. respectively.

The special biological refrigeration chart is for seven days and covers the temperature range —5 C. to +15 C.
(Manufacturers: Shandon Scientific Co., Ltd., 6 Cromwell Place, London, S.W.7).

Ultra Violet Bench Light

Designed around the new miniature 9 in. ultra violet fluorescent tubes is a recently marketed bench light.

This is for use on desk or bench and has many applications among others for engineering inspection, printers, dyers, forensic and medical laboratories, gemmologists and geologists, textile manufacturers, etc.

The light source is two 9 in. miniature fluorescent tubes giving radiation in the 3650 A.U. There is no radiation below 3100 A.U. and almost no visible light.

Always ready for use without delay for warming up the lamp is very efficient with high U.V. output for a consumption of only 20 watts. The tubes run quite cool and have an average life of 5,000 hours.

The lamp housing is fully adjustable including vertical positioning and the whole unit is stable in any position on the heavy 7 in. diameter base.

A separate electro brightened aluminium reflector is fitted to ensure maximum radiation and a 4 m/m. 'Woods' glass screen to filter out radiation in the visible waveband.

Supplied complete with tubes and connecting cable the lamp is for use on 200-250 volt A.C. mains only.
(Manufacturers: P. W. Allen & Company, 253 Liverpool Road, London, N.1).

Allen A.409 bench type ultra violet light.

New Sensitive Null Indicator

A new instrument, the equivalent of a very sensitive galvanometer but without the disadvantages of a conventional galvanometer, has been introduced. It is not affected by vibration and it comes to rest very quickly.

For use anywhere within reach of a mains supply, the new instrument is available as a single unit weighing 15 lb. only; it measures approximately 8 in. × 5 in. × 17 in. overall.

This null indicator does away with the disadvantages of a conventional galvanometer.

Use of the instrument is envisaged in laboratories, colleges, development departments, hospitals and the like; also for any industrial inspection or testing routines which include a bridge measurement. As it is not sensitive to vibration or level, it makes an ideal unit for testing at the end of a production line. A very practical advantage of the indicator is that no resistors are needed for protection, since a temporary overload of up to 1½ volts will do no harm.

Designated ElectroniK Null Indicator 104W1-G, the instrument is available for 115 volts 50 cycles, price £100, or for 200-250 volts 50 cycles (with built-in mains transformer), price £103.
(Manufacturers: Honeywell Controls Ltd., Greenford, Middlesex.)

Ultrasonic Cleaners

Recent additions to the range of ultrasonic cleaners available in this country are the American DiSONtegrator Series, the smallest of which is the system 40, which has a tank capacity of ¼ gallon (U.S.).

The generator is operated by a simple push/pull frequency control. The operating frequency of this unit, and also the 1½ gallon (U.S.) System 80, is 90 Kc. The larger units which are available operate at 40 Kc.

The tanks are manufactured from deep drawn stainless steel. A choice of seven colours is available for the finish of the generator and tank case. Extra tanks and change over switches are also obtainable if more than one solvent or cleaning operation is necessary.
(Distributed by: Continental Distributors Ltd., 121 Earls Court Road, London, S.W.5, and Headland Engineering Developments Ltd. (Electro-Mechanical Division), 10 Melon Road, Peckham, London, S.E.15).

This ultrasonic homogenizer offers the easiest, most convenient and efficient way of making emulsions and dispersions.
Minisonic Model 3 is made in stainless steel with built-in glass vessels and perfect finger tip control valve.
Developed on the experience of over a thousand installations of the Rapisonic, it is an advanced and reliable machine, sold at a sensible price.
Details on request from *Ultrasonics Ltd.*, Westgate, Otley, Yorkshire.

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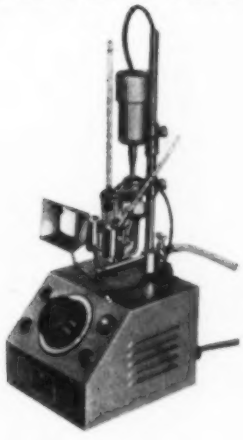

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NEWS

General

Course on Corrosion

A special one-year course on 'Corrosion and Protection of Metals' is to be held by the Borough Polytechnic, Borough Road, London, S.E.1.

This will be operated both as a part-time day (one afternoon and one evening) and as an evening course (two evenings a week) and at the end of it students will be able to sit for the Borough Polytechnic 'Corrosion Engineering Certificate' examination.

The syllabus covers the chemical, electro-chemical and metallurgical principles in addition to corrosion and protection, so that students should be in a position to approach corrosion problems with an adequate knowledge of the scientific principles involved.

Russian Visit to Cambridge

Prof. V. V. Parin, Director of the Institute for Natural Pathology and Physiology in Moscow, and three colleagues, Dr. V. V. Guzoff, Dr. K. P. Vezno, and Mr. V. A. Klukachol, recently visited Cambridge as guests of the Cambridge Instrument Company and the Department of Physiology at Cambridge University.

The professor, who is also a full time member of the U.S.S.R. Academy of Medical Sciences, spent some time in the company's research laboratories and factory where he and his colleagues were able to see working demonstrations of numerous medical, industrial and biological instruments, including some made by the company's new associate, Electronic Instruments Ltd. Prof. Parin was particularly interested in the Huxley ultra-microtome, an instrument used for cutting very thin sections of biological tissue for examination under electron-microscopes. Another instrument of interest was a fully transistorized portable cardiograph that can be used not only for routine work in hospitals and clinics, but also for on-the-spot diagnosis under difficult conditions, such as in the patient's home, in factories, or even on the side of a mountain.

Trypsinization process by which the monkey kidney tissue is separated into single cells—a stage in the production of oral polio vaccine.

Oral Polio Vaccine

Pfizer Ltd. announce that production of the new Sabin-type oral polio vaccine is already well advanced in Great Britain, and that the U.S. authorities have adopted the Sabin virus as a standard for manufacturing. This decision is likely to be followed by other governments, thus opening the way to widespread use of oral vaccine in many countries of the world.

After a successful and consistent performance in production of the Salk-type inactivated, injectable vaccine the company commenced manufacture of the Sabin-type product at the beginning of the year and already have millions of doses undergoing the arduous testing procedures shortly to be regularized by the U.S. authorities.

Pfizer Ltd. will supply its associate company in the U.S. with the new vaccine, thus enabling them to make it available there at an early date. All vaccine produced is also expected to meet British test regulations. The vaccine will, therefore, be available to the Ministry of Health, if required by them for vaccination programmes in Britain.

Mr. C. H. Rose, head of the medical department at the Cambridge works demonstrates the Electrite cardiograph and the electrocardioscope to the Russians.

Manufacturers' News

Griffin Balances

The sales of the Griffin 'Minor' balance, recently introduced for general students' use in schools, technical colleges and universities and also in industrial laboratories, have been such that the price has now been reduced from £7 5s. 0d. ex works, United Kingdom, per balance, to £6 12s. 6d.

This new series includes the 'Duorider', 'Chaindial', and 'Standard', developed by Griffin & George (Research and Development) Ltd., another member company of the Griffin and George Group.

Potassium Iodide

Interesting Facets

of chemical manufacture include the occurrence, from time to time, of unusual crystal "build-ups". This photograph was taken in one of the many May & Baker laboratories producing chemicals for use throughout the world. There are now more than 650 **M&B** brand laboratory chemicals available to cover the everyday requirements of academic, industrial, and research laboratories.

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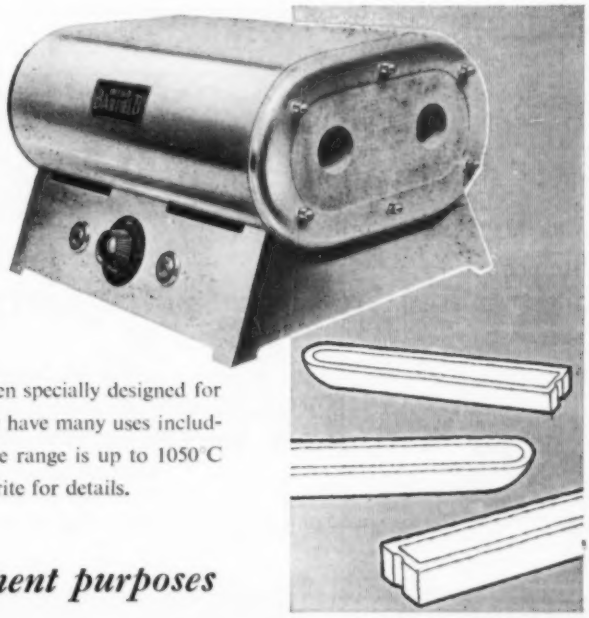

Wild-Barfield laboratory tube muffles have been specially designed for use in analytical and works laboratories. They have many uses including critical point determinations. Temperature range is up to 1050°C (1922°F). Models are available from stock. Write for details.

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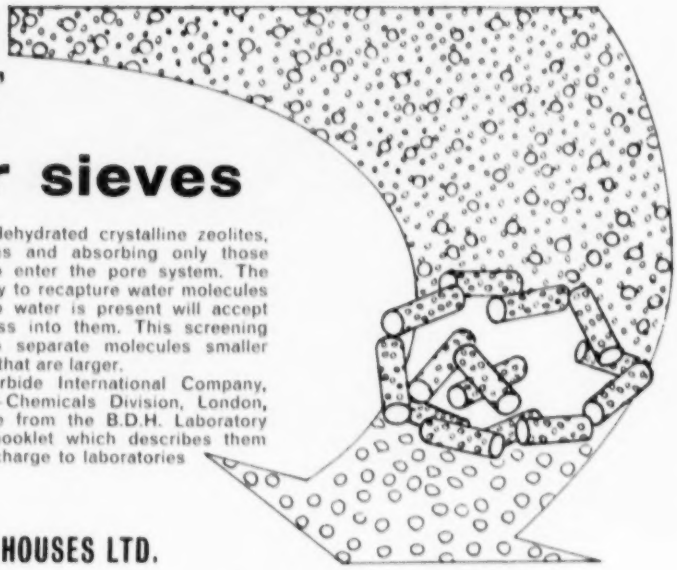

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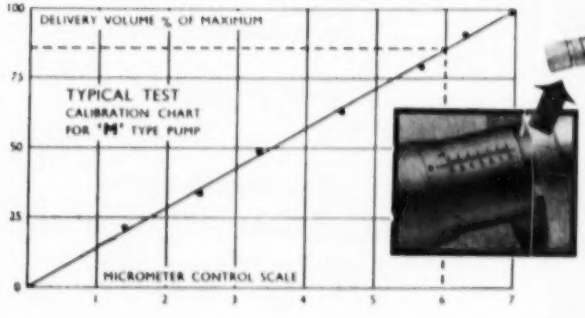

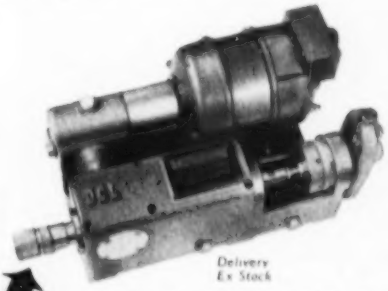

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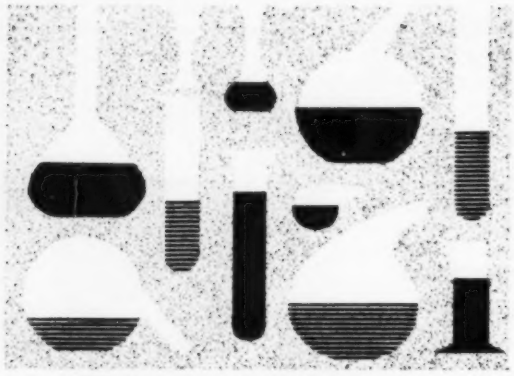

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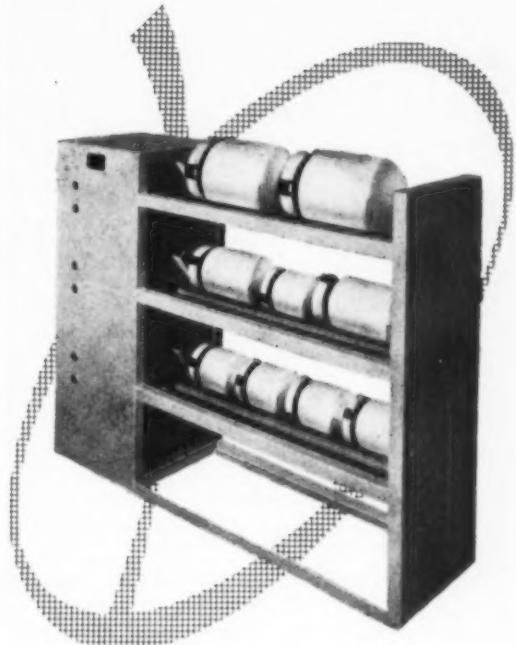

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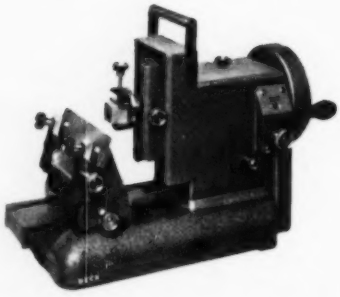

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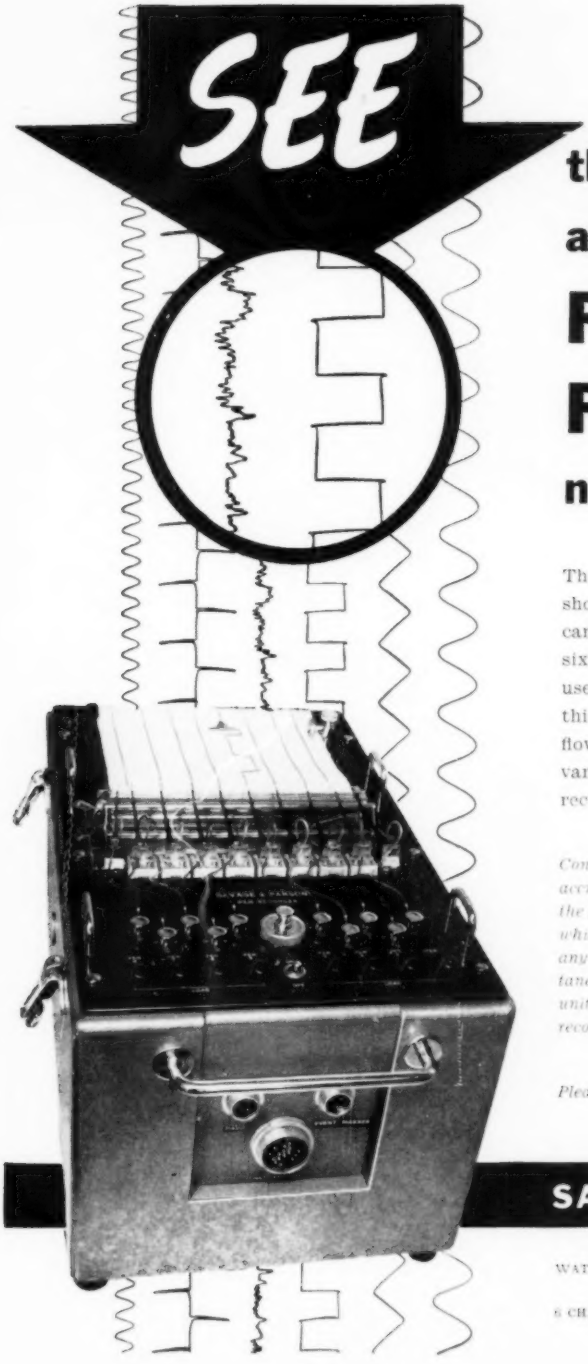

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